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CHARACTERIZATION AND EVALUATION OF RE-REFINED ENGINE LUBRICATING OIL

INTERIM REPORT
AFLRL No. 152

By

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**U.S. Army Fuels and Lubricants Research Laboratory
Southwest Research Institute
San Antonio, Texas**

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engine deposits derived from virgin and re-refined engine oils. (2) The effects of virgin and re-refined oils on engine blowby composition and engine deposit generation were determined using a spark ignition engine and, 3) Virgin and re-refined basestock production consistency were determined by participating in the ASTM/NBS cooperative basestock consistency study.

FOREWORD

The work reported herein was conducted at the U.S. Army Fuels and Lubricants Research Laboratory (USAFRLRL), located at Southwest Research Institute, San Antonio, TX, under Contract Nos. DAAK70-80-C-0001 and DAAK70-82-C-0001 during the period October 1979 through September 1981. The United States Department of Commerce, National Bureau of Standards (NBS) participated in the program under Interagency Agreement W26AAL-79303-001. Mr. Donald A. Becker was the NBS project officer, while Mr. T.C. Bowen, Jr. of the U.S. Army Mobility Equipment Research Command (USAMERADCOM, DRDME-GL) was the Army project officer.

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I. INTRODUCTION

More than two billion gallons of lubricating oils are used each year in the United States. Approximately 50 percent of this total is consumed or otherwise lost during use.^{(1)*} The remaining one billion gallons per year of used lubricating oil are a significant and valuable resource. With proper re-refining treatment, used oil can be utilized as a fuel, or, more importantly, it can be reused as a lubricant or lubricant basestock. The U.S. government has enacted important legislation in recent years to encourage the utilization of this valuable natural resource.⁽²⁾

In December 1975, the Energy Policy and Conservation Act (Public Law 94-163) was passed. This Act instructed the National Bureau of Standards (NBS) to develop test procedures to determine the potential "substantial equivalency" of recycled oil products to virgin oil products. NBS was required to transmit these test procedures to the Federal Trade Commission (FTC) for its use in developing trade regulation rules.

The Resource Conservation and Recovery Act (PL-94-580) of 1976 called for a review of Federal government specifications to allow for the purchase of products containing recycled materials. The U.S. Army responded to this by conducting research on the feasibility of making military engine oils from re-refined basestocks, which resulted in the revision of engine oil specification MIL-L-46152 to eliminate restrictions on re-refined oil use. Finally, the Model (States) Used Oil Recycling Act of 1980 (PL-96-463) assists states in establishing control over used oil disposal.

The current NBS-Army program was initiated to obtain additional data base on comparative performance of re-refined and virgin oils, and to investigate the potential "substantial equivalency" of re-refined and virgin oils. The program consisted of 1) the development of an engine deposit removal, recovery, and analysis methodology, which was used to compare engine deposits derived from virgin and re-refined engine oils, 2) a comparison of engine blowby and deposit generation tendencies of virgin and re-refined oils using

*Underscored numbers in parentheses refer to the list of references at the end of this report.

a single-cylinder spark ignition engine, and 3) participation in the ASTM-NBS cooperative basestock consistency study. Each of these phases of the overall program is discussed in the following sections of this report.

II. DEVELOPMENT OF DEPOSIT REMOVAL AND ANALYSIS METHODOLOGY USING 2.3-L ENGINE PARTS

Development of methodology for the removal and recovery of engine deposits initially focused on solvent removal of varnish deposits from Sequence VD engine parts. The recovered varnish deposits were then analyzed by a variety of instrumental and chemical techniques.

The cam baffle from the 2.3-L engine used in the Sequence VD test was selected for initial deposit removal because it was simple to wash and contained a reasonable amount of deposit. The entire cam baffle was sequentially washed with the following series of solvents of increasing polarity or solvency power:

1. Heptane
2. Toluene (T)
3. Methanol (M)
4. Acetone (A)
5. TAM combination (equal volumes)
6. Methylene chloride

Since the engine part surfaces are wetted with engine oil, it was decided to remove this oil separately from the deposits to simplify the analysis of the deposits. Heptane was chosen to remove this oil and any other readily soluble, low-polarity or low-molecular weight material. Toluene was chosen to dissolve the more polar or the higher molecular weight asphaltene materials. The increasingly polar methanol, acetone, and TAM (equal volumes of toluene/acetone/methanol) dissolved the more resinous, high-molecular weight deposits.

A final wash with methylene chloride removed the small amount of deposit

remaining after the TAM washing. The solvent from each collected wash was removed using a vacuum rotary evaporator. The collected deposit fractions were then analyzed for molecular weight and elemental content as shown in Table 1. While this washing procedure removed the deposit, it was judged to

TABLE 1. 2.3-L ENGINE CAM BAFFLE DEPOSIT ANALYSIS

Deposit Fraction*	Wt% of Recovered Soluble Mat'l	Data for Specific Soluble Fractions					
		Avg Mol Wt	% S	% N	C/H Ratio	% C	% H
Heptane Soluble	72	420	2.0	1.0	6.7	83.4	12.5
Toluene Soluble	15	900	4.4	6.0	8.8	67.8	7.7
Methanol Soluble	5	800	3.9	7.0	8.7	59.9	6.9
Acetone Soluble	7	1300	2.5	5.0	9.2	63.9	6.9
TAM Soluble	1	950	0.4	3.0	IS	IS	IS
Methylene Chloride Soluble	<1	1150	IS	4.0	IS	IS	IS

* = Engine part was washed sequentially with solvents; insoluble matter was not measured.

IS = Insufficient sample

be rather difficult and too complex. It also allowed possible contamination of fractions with insoluble matter loosened by the solvents. Thus, a modified procedure was developed which resulted in deposit removal from the engine part with a solvent mixture of equal volume parts of toluene/acetone/methanol (TAM). The TAM mixture completely removed all deposits found on the cam baffle, rocker cover, oil pump pickup tube and screen, and the front seal housing of the 2.3-L engine. This procedure was adopted to simplify the washing and filtration process and minimize contamination of fractions with insoluble matter. The TAM washings were filtered to remove any suspended insoluble or metallic particulate matter. The TAM solvent was removed with a rotary evaporator, and the soluble deposit was recovered and weighed. The collected deposit was then sequentially extracted with n-heptane, toluene, and finally, TAM. Each of these fractions was then recovered using solvent removal by the vacuum rotary evaporator. The recovered individual deposit fractions were then analyzed as shown in Table 2. Next, the deposits from

TABLE 2. 2.3-L ENGINE DEPOSIT ANALYSIS

<u>Deposit Fraction</u>	<u>Wt% of Recovered Soluble Mat'l</u>	<u>Data for Specific Soluble Fractions</u>					
		<u>Avg</u>	<u>C/H</u>			<u>% C</u>	<u>% H</u>
		<u>Mol Wt</u>	<u>% S</u>	<u>% N</u>	<u>Ratio</u>		
<u>Rocker Arm Cover</u>							
Heptane Soluble	69	420	1.1	1.0	6.7	82.9	12.3
Toluene Soluble	19	770	1.8	3.0	9.1	67.9	7.5
TAM Soluble	12	1470	1.9	3.0	9.2	62.3	6.8
<u>Oil Pump Tube</u>							
Heptane Soluble	59	400	1.2	1.0	6.8	81.7	12.0
Toluene Soluble	31	635	1.4	3.0	8.6	68.4	8.0
TAM Soluble	10	1625	1.4	5.0	9.9	60.8	6.2
<u>Oil Pump Screen</u>							
Heptane Soluble	78	400	1.0	1.0	6.8	80.3	11.8
Toluene Soluble	10	560	1.5	2.0	8.3	70.4	8.4
TAM Soluble	12	1900	2.0	3.0	9.7	60.5	6.2
<u>Timing Gear Cover</u>							
Heptane Soluble	54	410	IS	1.0	6.8	81.7	12.0
Toluene Soluble	27	610	IS	2.7	8.9	68.8	7.7
TAM Soluble	19	IS	IS	2.4	7.3	61.7	8.4

IS = Insufficient sample
Insoluble matter was not measured.

2.3-L engine pistons were removed and recovered. The piston skirt, oil rings and oil ring grooves were washed with TAM. The resulting deposit was extracted with heptane, toluene, and TAM as before. Finally, the piston crown, compression rings, lands and grooves were washed with N,N-dimethylformamide (DMF). Based on the work of Harris, et al. (3), DMF was selected as a more effective varnish removal solvent than TAM. The DMF washings were filtered through a Whatman No. 541 filter to remove any suspended insoluble or metallic particulate matter. DMF was removed using a vacuum rotary evaporator, and the recovered deposit was solvent extracted with sequential washes of heptane, toluene, TAM, and DMF. The analyses of the piston area deposit fractions are presented in Table 3.

TABLE 3. 2.3-L ENGINE DEPOSIT ANALYSIS FOR PISTON AREA

<u>Deposit Fraction</u>	Wt% of	<u>Data for Specific Soluble Fractions</u>					
	Recovered	Avg	C/H				
	Soluble	Mol Wt	% S	% N	Ratio	% C	% H
	Mat'l						

* = Beyond scope of method for DMF soluble
 IS = Insufficient sample
 Insoluble matter was not measured.

While the data presented in Tables 1 through 3 were obtained primarily to aid in methodology development, some interesting trends concerning the chemistry of Sequence VD deposits were revealed. As expected, by using increasingly polar solvents, the extracted deposit materials were separated by polarity and complexity. It is of interest to note that deposits from the lower temperature engine areas such as oil pump screen and rocker arm cover had higher molecular weight polar deposits and had higher nitrogen and sulfur contents than the deposits from the higher temperature piston areas. This could be due to thermal cracking of large molecules in the higher temperature piston areas.

Based on the 2.3-L engine deposit analysis work, a deposit removal, recovery, and analysis methodology was finalized. This methodology is described in detail in the following section.

III. FINALIZED DEPOSIT METHODOLOGY

A. Piston Deposit Removal Procedure

The piston deposits are removed in three steps. A mixture of TAM and DMF is used to wash the piston, which removes essentially all deposits. The first step is to remove deposits from piston skirts, oil rings, and oil ring grooves with the wash mixture. Secondly, the piston crown, compression ring, land, and groove deposits are removed. The third step is to remove deposits from the undercrown portion of the piston. Figure 1 illustrates the piston wash areas. Washing the parts in this order allows good separation of

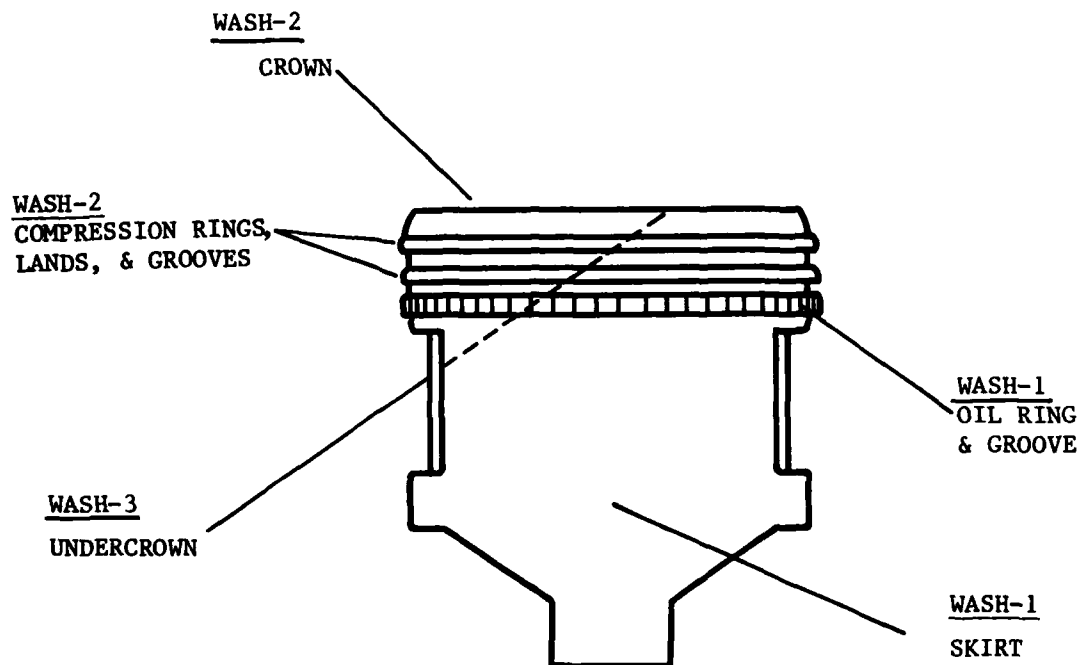


FIGURE 1. PISTON WASH AREAS

deposits from the separate areas of the piston, as the wash is never allowed to flow across unwashed areas. The mixture of TAM and DMF allows simultaneous removal of both oily deposits and high molecular-weight polar residues. Insoluble materials are also removed, suspended in the wash mixture.

B. Piston Deposit Recovery Procedure

Washes from the three different areas of the piston are collected separately and subjected to a recovery procedure. A flow chart illustrating the engine part washing and deposit recovery procedure is shown in Figure 2.

The recovery procedure involves filtering through a Whatman No. 541 filter, then evaporating the TAM/DMF wash mixture for a given piston area on a vacuum rotary evaporator. The residue remaining in the evaporator is then agitated with heptane, in order to remove oils and nonpolar materials. The heptane solution is filtered through a Whatman No. 43 filter and heptane is evaporated off with the rotary evaporator, producing the heptane-soluble fraction of the deposits.

The wash residue is then agitated with toluene in order to remove asphaltenes, and the toluene solution filtered through the Whatman No. 43 filter and evaporated to produce a toluene-soluble fraction. The same procedure is followed with TAM in order to isolate resins in a TAM-soluble fraction, and with DMF to solubilize high-molecular weight polar residues. Each solvent wash is continued until the solvent coming through the filter appears colorless. No more than 4 liters of TAM/DMF mixture, no more than 2 liters of the other solvents were used. The residue remaining in the No. 541 filter used to filter the original TAM/DMF part washing mixture for the piston area, and the No. 43 filter used to filter solutions during fractionation, form the insoluble residue fractions. These fractions are composed of metals and insoluble carbonaceous materials.

C. Analysis of Deposits

Each deposit fraction from each of the three piston areas was analyzed for carbon, hydrogen, nitrogen, sulfur, and qualitative elemental content. An infrared scan was made on each fraction, and on soluble fractions, molecular weights were determined. Quantitative elemental analysis was performed on insoluble fractions.

An infrared spectrum was obtained for each deposit fraction using a Beckman

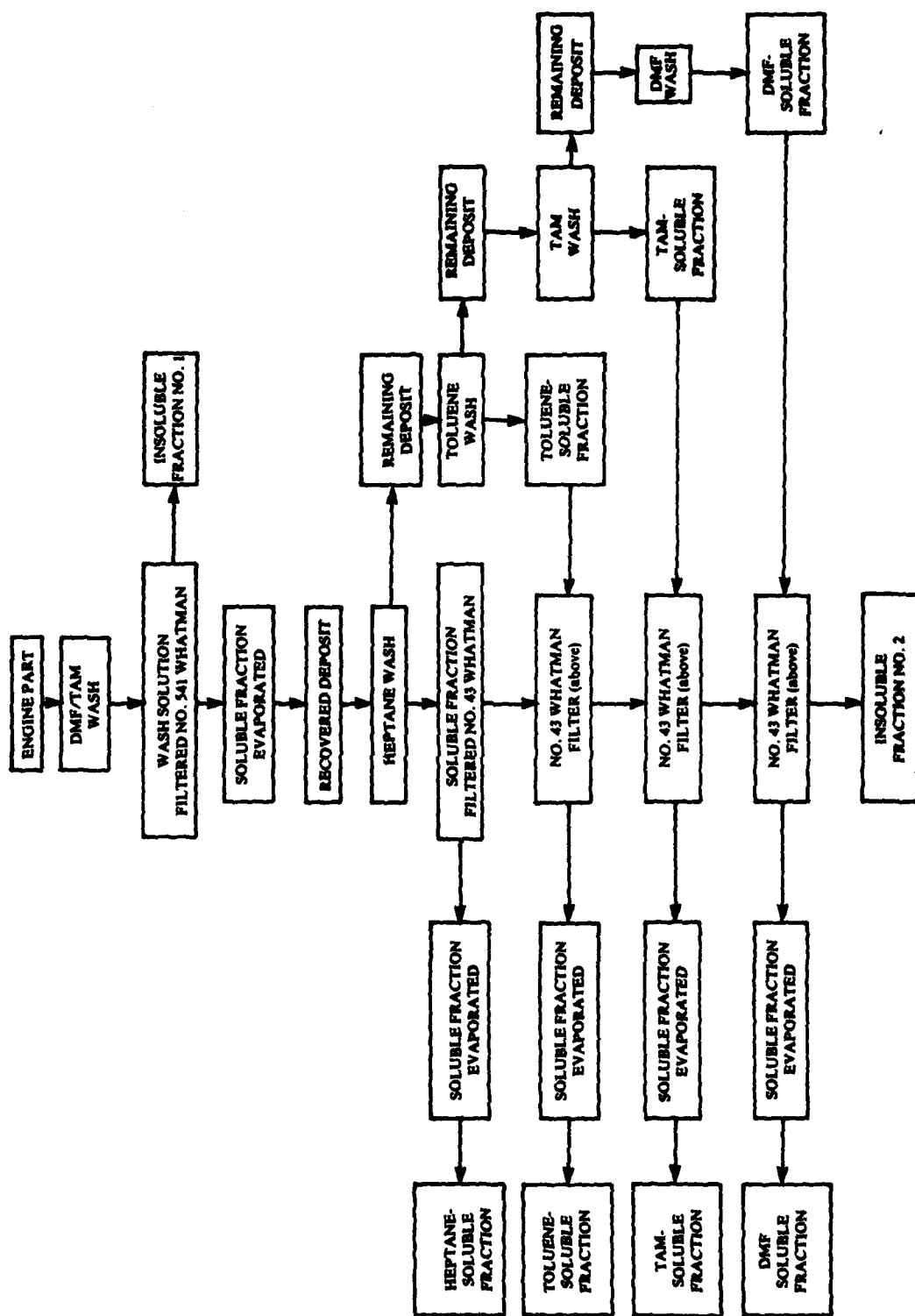


FIGURE 2. ENGINE PART WASHING AND DEPOSIT RECOVERY PROCEDURE

Microlab 620 MX Computing Spectrophotometer. When the fraction was a solid material, spectra were obtained using a 13-mm KBr pellet formed in a vacuum die. For fluid fractions, spectra were obtained using a demountable window holder. A small drop of the fraction was placed between NaCl windows in the holder, and the resulting thin film was scanned.

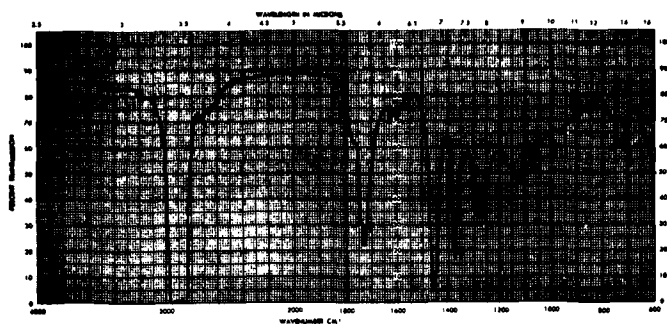
Special attention was given to absorption bands around 1710 cm^{-1} , 1640 cm^{-1} , and 1550 cm^{-1} , as these give evidence of oxidation and nitration. The band near 1710 cm^{-1} is due to the carboxyl group (C=O); bands near 1640 cm^{-1} to nitro compounds, and bands near 1550 cm^{-1} to carboxylate salts and nitro compounds. Typical spectra for representative fractions are shown in Figure 3. Each of these fractions shows a fairly large absorption near 1710 cm^{-1} , showing oxidation. The infrared spectra were used to note the presence or absence of absorption due to oxidation and nitration in fractions and to observe trends. Due to small sample sizes and varied methods of sample preparation from fraction to fraction, no quantitative work was attempted on these samples. Use of infrared studies in the program is discussed further in the section on CLR engine tests for virgin and re-refined basestock oils.

An EDAX International EXAM (Elemental X-ray Analysis of Materials) System and an EDAX model 707B Energy Dispersive X-ray Analyzer were used to make sulfur determinations and qualitative elemental determinations. Sulfur was determined by dissolving fractions in TAM or DMF and comparing with sulfur standards in these solvents. Qualitative elemental analysis was performed directly on fractions using no solvents.

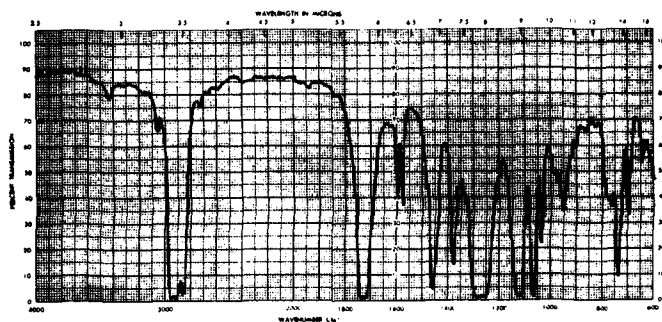
Carbon and hydrogen analysis was performed according to ASTM D 3178 Carbon and Hydrogen in the Analysis Sample of Coal and Coke. Nitrogen was determined using an Antek instrument based on the chemiluminescence principle. Molecular weight was determined by vapor osmometry, using chloroform or DMF as the solvent. Since samples must be dissolved for this method, molecular weight determinations were not made on insoluble fractions.

D. Briquetting Method Development

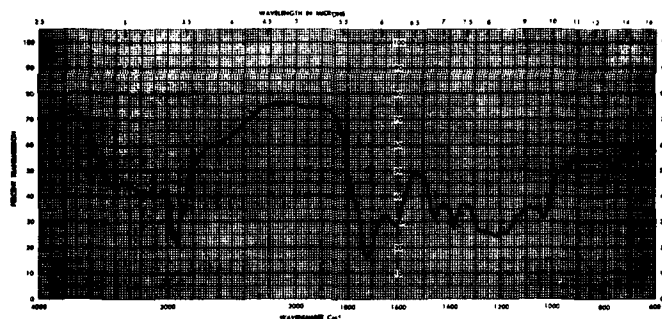
To better define insoluble residues in engine deposits, a briquetting method



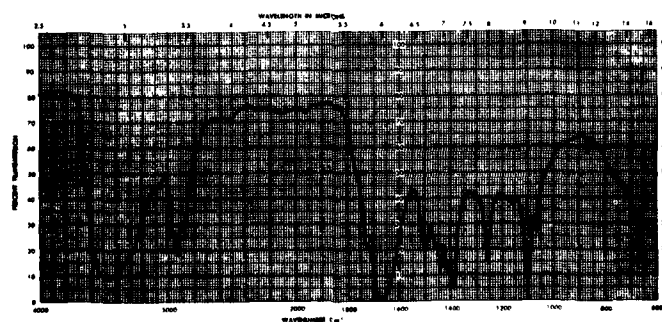
a. Heptane Fraction



b. Toluene Fraction



c. TAM Fraction



d. DMF Fraction

FIGURE 3. REPRESENTATIVE IR SPECTRA

has been developed for determining elemental content. This method employs an energy-dispersive X-ray fluorescence spectrometer. An advantage of energy-dispersive X-ray fluorescence analysis is the ability to simultaneously analyze for virtually every element with an atomic number greater than that of sodium. The method may not be used to analyze for C, N, or O.

The briquetting method requires the mixing of a ground engine deposit residue with a plasticizing binder followed by pressing into a sample pellet and X-ray fluorescence analysis of the pellets. The briquetting method was chosen as a means of analyzing samples which cannot be put into solution without the errors involved in loose powder analysis, in which a reproducible measurement is difficult to obtain.

Preparation of sample involves a preliminary grinding with a mortar and pestle to ensure a reasonably homogeneous sample. This is followed by grinding a mixture of Somar-mix additive and sample, in approximately a 4:1 ratio, in a vibrating ball mill grinder. The Somar-mix is an organic, chemically inert substance which will not chemically react with sample materials or introduce any interfering X-ray spectra. The lubricious and abrasive constituents in the mix intermix with the sample during comminution, uniformly reduce sample size, and produce a homogeneous blend. The additive also serves as a briquetting agent, forming materials into "plasticized" sample pellets which are firmly bonded together to uniform densities and which have smooth, unblemished surface finishes for statistically reproducible results and minimized intensity variations. Pellets are formed using compressible aluminum X-ray pellet cups in a 1.25-in. mold assembly. In order to avoid thin, brittle pellets due to the small sample size used, the pellet cup is first filled two-thirds full with Somar-mix and lightly pressed. The sample and Somar-mix mixture is then placed on the pellet, and the sample is pressed at 25,000 pounds total load on a hydraulic press. The resulting pellet is about 3/16 in. thick with a smooth, highly plasticized surface.

In the X-ray spectrochemical analysis of powdered materials, factors such as matrix composition, interelement effects, absorption, and enhancement play important roles in the analytical accuracy of elemental determinations. In

order to treat these factors, an XRF regression program has been used in analysis of the samples. Standards were prepared using metal oxides or metal hydroxides in similar proportions to those observed in qualitative examination of the deposits. Interelement effects and matrix composition can be taken into account in the XRF regression program and concentration vs counts per second calibration curves formed for each element observed. Samples may be analyzed, and their counts per second entered in the program to produce weight percent data.

The previously described deposit analysis methodology was used to examine and compare laboratory- and field-derived engine deposits for both virgin and re-refined engine oils, as described in the following section.

IV. COMPARISON OF ENGINE DEPOSITS FROM VIRGIN AND RE-REFINED ENGINE OILS

A. Introduction

Re-refined and virgin basestock engine oils have completed a field evaluation by the Royal Canadian Mounted Police (RCMP). The RCMP vehicles were 1979 model sedans equipped with police-duty 5.0 liter (305-cubic inch) displacement V-8 engines and automatic transmissions. The vehicles were based in Ottawa, Canada and used around the clock for city-driving type patrol service.(4) The two formulated engine oils evaluated by the RCMP each contained the same additive package and treatment level. Table 4 contains the properties of the base oils and formulated engine oils tested in this program.(4)

The formulated engine oils used in the RCMP field test were also evaluated in standard ASTM laboratory engine tests. The L-38, Sequence IID, and Sequence IIID, engine test results for the virgin-based engine oil (FLO 79038) and the re-refined engine oil (FLO 79034) are presented in Table 5 and compared to the requirements for API service classifications SE and SF.(5)

B. Deposit Analysis

Pistons from both field service and laboratory testing were subjected to the deposit analysis methodology described in the previous section, to determine

if virgin and re-refined engine oils formed equivalent engine deposits. The details of the deposit comparison matrix are shown in Table 6. The deposit analysis data are presented in Tables 7 to 11. Due to small sample size of some fractions, all analyses could not be performed on every fraction. Insufficient sample is reported where test data are omitted for this reason.

TABLE 4. PROPERTIES OF BASE OILS AND FORMULATED OILS

Property	ASTM No. or Procedure	Re- Refined Base Oil	Virgin Base Oil	Formulated Re- Refined Oil FLO 79034	Formulated Virgin Oil FLO 79038
Viscosity @ 100°C, cSt	D 445	7.51	7.30	13.90	13.70
Viscosity @ 40°C, cSt	D 445	52.62	53.43	105.50	107.10
Viscosity Index	D 2270	104.5	95	132.5	127.5
Gravity, API	D 287	29.6	29.7	28.5	27.9
ASTM Color	D 1500	L 3.0	L 1.0	4.0	2.5
Pour Point, °C (°F)	D 97	-12 (10)	-7 (20)	-1 (30)	-3.8 (25)
Flash Point, °C (°F)	D 92	215 (420)	224 (435)	227 (440)	229 (445)
Cloud Point, °C (°F)	D 2500	-9 (16)	-7 (20)	-9 (16)	-8 (18)
Fuel Dilution, Vol%	D 322	0.4	0.6	0.6	0.4
	D 3525	0.03	0.02	0.03	0.02
Total Acid No.	D 664	0.10	0.02	2.5	2.24
Total Base No.	D 664	0.00	0.02	5.74	5.71
Strong Acid No.	D 64	NIL	NIL	NIL	NIL
Initial pH	D 64	7.0	6.6	6.9	7.2
Saponification No.	D 94	0.25	0.18	ND	ND
Ramsbottom Carbon Residue, wt%	D 524	0.17	0.09	1.14	1.07
Total Ash, wt%	D 482	0.002	0.000	1.05	1.04
Uncoag. Pentane Insolubles, wt%	D 893/A	0.001	0.000	0.001	0.003
Copper Corrosion, 3 hr @ 212°F	D 130	No. 1	No. 1	No. 1	No. 1
Total Solids, mg/100 ml	D 2276	1.64	0.76	18.76	15.84
Aniline Point, °C (°F)	D 611	106 (223)	107 (225)	ND	ND
Hydrocarbon Types, wt	D 2007				
Saturates		76.1	81.0	73.48	75.0
Polar Compounds		2.1	0.9	5.7	5.1
Aromatics		21.8	18.1	20.4	19.9

ND = Not determined

TABLE 4. PROPERTIES OF BASE OILS AND FORMULATED OILS (continued)

Property	ASTM No. or Procedure	Re- Refined Base Oil	Virgin Base Oil	Formulated Re- Refined Oil	Formulated Virgin Oil
Elemental Content, ppm	Spectro- chemical analysis; AA				
Calcium		<10	<10	2200	2200
Barium		<10	<10	1	4
Zinc		2	<1	1400	1400
Magnesium		<1	<1	6	8
Sodium		<1	<1	3	14
Lead		<1	<1	1	12
Silicon		<5	<5	3	3
Iron		1	<1	1	1
Copper		<1	<1	1	1
Aluminum		<1	<1	1	2
Chromium		<1	<1	1	1
Manganese		<1	<1	ND	ND
Nickel		<1	<1	ND	ND
Total Sulfur Content, wt%	D 129	0.19	0.09	ND	ND
Total Nitrogen Content, ppm	microcoul	6	56	ND	ND
Glycol Test	D 2982	Neg.	Neg.	ND	ND
Foam Test	D 892				
Foaming Tendency @ 75°F		440 ml	410 ml	0 ml	0 ml
Foaming Tendency @ 200°F		40 ml	40 ml	50 ml	30 ml
@ 75°F after test @ 200°F		380 ml	490 ml	0 ml	0 ml
Foam stability @ 75°F		N11 after 7 min.	N11 after 8 min.	N11	N11
Foam stability @ 200°F		N11 after 30 sec.	N11 after 3 sec.	N11 after 30 sec.	N11 after 25 sec.
@ 75°F after test @ 200°F		N11 after 7 min.	N11 after 9 min.	N11	N11

ND = Not determined

The deposit weight distribution by piston area is shown in Table 12 for each group of pistons which were washed. The deposit weight distribution was nearly the same for virgin and re-refined oils from a given source (e.g., lab or field). In all cases, the undercrown had the least deposit weight. For field-use pistons, the crown area had the most deposit weight while the skirt

TABLE 5. LABORATORY ENGINE TESTS

	SE	SF	FLO 79038 VIRGIN OIL	FLO 79034 RE-REFINED OIL
L-38, Bearing weight loss, mg	40 max	40 max	31.5	22.4
Sequence IID				
Average engine rust rating	8.5 min	8.5 min	8.43 ^a	8.55
Lifter sticking	None	None	None	None
Sequence IIID @ 64 hr ^d			#1	#2
Visc. Inc. @ 40 °C, %				
After 40 hr	375 max	-	48	47
After 64 hr	-	375 max	1875 ^b	1000 ^b
Avg. engine ratings @ 64 hr				
Sludge	9.2 min	9.2 min	9.45	9.3
Piston skirt varnish	9.1 min	9.2 min	8.8 ^c	8.7 ^c
Oil ring land deposits	4.0 min	4.8 min	7.4	6.7
Ring sticking	None	None	None	One ^c
Lifter sticking	None	None	None	None
Scuffing & wear @ 64 hr				
Cam or lifter scuffing	None	None	None	None
Cam plus lifter wear, μm				
Average	102 max	102 max	45.7	58.4
Maximum	254 max	203 max	96.5	101.6

^a Fails SE requirement

^b Fails SF requirement

^c Fails both SE and SF requirements

^d Sequence IIID test performed in duplicate on virgin oil

TABLE 6. TEST MATRIX LUBRICANTS

	FLO 79038 VIRGIN	FLO 79034 RE-REFINED
Field Test	Cell A Vehicle Nos. 211, 213, 215, 217	Cell B Vehicle Nos. 212, 214, 216, 218
Lab Eng Test	Cell C III-D test #21-180	Cell D III-D test #21-179

TABLE 7. ANALYSIS OF HEPTANE-SOLUBLE PISTON DEPOSITS

Analysis	Piston Skirts (Heptane Fraction)				Piston Undercrowns (Heptane Fraction)				Piston Crowns (Heptane Fraction)			
	III D 21-179 (re-refined)	III D 21-180 (virgin)	Field Test (virgin)	Field Test (virgin)	III D 21-179 (re-refined)	III D 21-180 (virgin)	Field Test (re-refined)	Field Test (virgin)	III D 21-179 (re-refined)	III D 21-180 (virgin)	Field Test (re-refined)	Field Test (virgin)
Qualitative Elements	-	-	-	-	Cl	Cl	-	Cl	Cl	Cl	-	-
Fraction, wt% of Soluble Deposit	74.9	87.2	56.4	65.9	72	56	18	47	70	56	5	5
C, wt%	83.6	83.3	84.7	84.4	*	82.70	*	84.51	83.16	83.14	85.71	84.82
H, wt%	12.9	12.7	13.1	13.1	*	13.02	*	13.16	12.82	12.63	13.23	13.20
N, wt%	0.76	0.23	0.26	0.31	0.27	0.47	0.19	0.46	0.25	0.24	0.17	0.29
S, wt%	0.22	0.28	<0.01	0.08	<0.01	0.13	<0.01	0.22	0.21	0.22	<0.01	0.18
MM, Avg	460	500	480	490	420	490	440	420	460	520	480	650
C/H	6.5	6.6	6.5	6.4	*	6.4	*	6.4	6.5	6.6	6.5	6.4

* = Insufficient Sample

- = None detected

TABLE 8. ANALYSIS OF TOLUENE-SOLUBLE PISTON DEPOSITS

Analysis	Piston Skirts (Toluene Fraction)				Piston Undercrowns (Toluene Fraction)				Piston Crowns (Toluene Fraction)			
	III D 21-179 (re-refined)	III D 21-180 (virgin)	Field Test (virgin)	Field Test (virgin)	III D 21-179 (re-refined)	III D 21-180 (virgin)	Field Test (re-refined)	Field Test (virgin)	III D 21-179 (re-refined)	III D 21-180 (virgin)	Field Test (re-refined)	Field Test (virgin)
Qualitative Elements	Cl	Cl	-	-	Cl, Zn	Cl	-	Cl	Cl	Cl	Zn	-
Fraction, wt% of Soluble Deposit	2	6	10	3	8	31	19	5	4	14	8	5
C, wt%	*	68.27	84.20	*	*	60.50	84.64	*	*	70.55	84.97	84.67
H, wt%	*	8.75	12.70	*	*	8.35	12.96	*	*	9.05	12.71	13.01
N, wt%	*	1.5	0.72	2.3	2.38	*	0.31	1.70	1.15	0.66	0.28	0.32
S, wt%	<0.01	0.82	0.14	0.65	1.91	0.08	<0.01	0.87	0.93	0.39	<0.01	0.21
Mt, Avg	*	340	460	*	*	470	440	*	570	440	440	400
C/H	*	7.8	6.6	*	*	7.3	6.5	*	*	7.8	6.7	6.5

* = Insufficient sample
- = None detected

TABLE 9. ANALYSIS OF TAM-SOLUBLE PISTON DEPOSITS

Analysis	Piston Skirts (TAM Fraction)				Piston Undercrowns (TAM Fraction)				Piston Crowns (TAM Fraction)			
	III D 21-179 (re-refined)	III D 21-180 (virgin)	Field Test (re-refined)	Field Test (virgin)	III D 21-179 (re-refined)	III D 21-180 (virgin)	Field Test (re-refined)	Field Test (virgin)	III D 21-179 (re-refined)	III D 21-180 (virgin)	Field Test (re-refined)	Field Test (virgin)
Qualitative Elements	Zn, Br	Cl	Zn, Cl	Zn, Cl	Cl	Cl	Zn, Cl	An, Cl	Zn, Cl	Zn, Cl, Br	Zn	Zn, Cl, M
Fraction, wt% of Soluble Deposit	1	2	17	18.5	11	7	16	36	3	9	50	42
C, wt%	*	*	60.05	59.31	*	*	59.18	53.77	*	*	78.42	68.44
H, wt%	*	*	6.15	6.46	*	*	6.07	6.45	*	*	10.57	7.64
N, wt%	*	*	3.06	1.7	2.70	*	2.35	3.28	1.17	2.2	2.40	3.29
S, wt%	0.01	1.68	1.80	0.97	0.01	0.85	2.48	1.77	0.01	0.96	0.39	1.90
MM, Avg	*	440	180	640	*	*	500	83	*	240	470	220
C/H	*	*	9.8	9.2	*	*	9.8	8.3	*	*	7.4	9.0

* = Insufficient sample

- = None detected

TABLE 10. ANALYSIS OF DMF-SOLUBLE PISTON DEPOSITS

Analysis	Piston Skirts (DMF Fraction)				Piston Undercrowns (DMF Fraction)				Piston Crowns (DMF Fraction)			
	III D 21-179 (re-refined)	III D 21-180 (virgin)	Field Test (virgin)	Zn	III D 21-179 (re-refined)	III D 21-180 (virgin)	Field Test (virgin)	Zn	III D 21-179 (re-refined)	III D 21-180 (virgin)	Field Test (re-refined)	Field Test (virgin)
Qualitative Elements	Pb, Br, Cl	Pb, Br, Cl	Zn	Zn	Pb, Br, Cl	Pb, Br, Cl	Zn	Zn	Pb, Br, Cl	Pb, Br, Cl	Zn	Zn, Mn
Fraction, wt% of Soluble Deposit	22	5	17	12.52	9	6	48	12	24	21	37	48
C, wt%	*	*	53.42	52.40	*	*	50.06	55.40	*	*	55.25	49.40
H, wt%	*	*	5.88	6.44	*	*	5.53	5.74	*	*	5.42	7.31
N, wt%	*	1.44	0.79	2.86	0.38	*	3.02	2.34	0.13	*	1.41	3.75
S, wt%	<0.01	5.92	0.65	0.92	<0.01	5.95	0.53	2.76	<0.01	7.18	6.24	0.86
Mn, Avg	*	530	+	*	400	*	+	+	580	340	+	330
C/H	*	*	9.1	8.1	*	*	9.1	9.7	*	*	10.2	6.8

* = insufficient sample
+ = insoluble in DMF

TABLE 11. ANALYSIS OF 541 FILTER (INSOLUBLES) FROM PISTON DEPOSITS

Quantitative Analyses, %	Piston Skirts (Filter #541)				Piston Undercrowns (Filter #541)				Piston Crowns (Filter #541)			
	III D 21-179 (re-refined)	III D 21-180 (virgin)	Field Test (re-refined)	Field Test (virgin)	III D 21-179 (re-refined)	III D 21-180 (virgin)	Field Test (re-refined)	Field Test (virgin)	III D 21-179 (re-refined)	III D 21-180 (virgin)	Field Test (re-refined)	Field Test (virgin)
P	-	-	3.8	1.7	*	*	2.0	3.7	-	-	3.8	3.7
S	6.7	6.0	10.3	3.5			4.3	4.1	6.2	6.4	3.7	6.3
Ca	0.5	0.4	9.3	4.5			4.3	4.8	0.4	0.5	4.4	3.4
Fe	7.5	3.4	-	0.3			-	-	3.9	4.3	0.5	-
Mn	-	-	0.7	4.1			4.2	1.5	-	-	4.6	10.1
Zn	0.5	0.2	10.9	2.6			2.0	3.2	0.3	0.3	2.1	2.2
Pb	24.2	13.0	1.0	0.4			0.8	-	17.2	19.2	0.8	3.0
Br	0.5	0.1	-	-			-	-	0.1	0.2	-	-
C	3.8	25.0	41.4	40.7			36.0	36.2	6.3	22.9	40.4	32.8
H	1.2	3.7	4.7	5.9			3.8	4.8	0.75	2.8	3.5	2.8
N	0.06	0.7	1.9	1.2			1.5	1.03	0.14	1.0	0.6	1.21
Total	45.0	52.5	84.0	64.9			58.9	59.3	35.3	57.6	64.4	65.5

* = Insufficient sample
 - = None detected

TABLE 12. DEPOSIT DISTRIBUTION BY PISTON AREAS
WT%

	<u>Re-refined Oil</u>		<u>Virgin Oil</u>	
	<u>Pistons</u>		<u>Pistons</u>	
	<u>Field</u>	<u>Lab</u>	<u>Field</u>	<u>Lab</u>
Number of Pistons washed	16	4	8	4
<u>Skirt</u>	36.9 (8.8534g)	42.3 (1.2300g)	30.1 (3.0582g)	47.5 (1.8690g)
<u>Undercrown</u>	6.0 (1.4449g)	15.9 (0.4608g)	6.7 (0.6789g)	9.9 (0.3892g)
<u>Crown</u>	57.1 (13.6860g)	41.8 (1.2170g)	63.2 (6.4271g)	42.6 (1.6775g)
<u>Total</u>	23.9843g	2.9078g	10.1642g	3.9357g
Avg deposit wt/piston	1.499g	0.727g	1.271g	0.984g

area of the lab engine pistons had the most deposit weight. Also, the average deposit weight per piston was highest for the field pistons. A further breakdown of deposit weight distribution by piston area and deposit solubility (solvent fraction) is shown in Table 13. Bar chart graphical presentations of the data presented in Table 13 are included as Appendix A. The lab engine pistons had much more heptane-soluble deposit for all three piston areas which is probably indicative of less engine oil drain-off from these parts due to less handling. Some differences were observed for re-refined (R) and virgin (V) oils. In lower temperature areas (skirt and undercrown), R oil tended to have higher deposit weights in the DMF fraction. Finally, for all oils, the field-derived deposit had much more TAM-soluble fraction than the lab-derived deposit.

TABLE 13. DEPOSIT DISTRIBUTION BY PISTON AREA AND FRACTION SOLVENT
WT%

Fraction	Skirt				Undercrown				Crown			
	Re-refined		Virgin		Re-refined		Virgin		Re-refined		Virgin	
	Field	Lab	Field	Lab	Field	Lab	Field	Lab	Field	Lab	Field	Lab
Heptane	56	74	66	87	18	72	47	56	5	70	5	56
Toluene	10	2	3	6	19	8	5	31	8	4	5	14
TAM	17	1	18	2	16	11	36	7	50	3	42	9
DMF	17	22	13	5	48	9	12	6	37	24	48	21
TOTAL	100	100	100	100	100	100	100	100	100	100	100	100

Some general overall trends were observed concerning the deposit analysis data from Tables 7 to 11. For all cases, the C/H ratio is highest for the heptane-soluble fraction and decreases as the deposit fractions increase in polarity. The nitrogen contents tended to be lowest in the heptane fraction and higher in the more polar fractions. From IR analyses, the content of carbonyl groups also tended to be higher in the more polar fractions as would be expected. Finally, the qualitative elements strongly reflect the fuel used (e.g., lead, bromine, and chlorine are present in lab engine deposits where leaded gasoline was used).

Next, the deposit analysis data were closely examined to determine if virgin and re-refined oil deposits had similar composition. Based on the test matrix of Table 6, the following comparisons of deposit composition were made:

- Virgin oil versus re-refined oil in field service.
(Cell A vs Cell B) as shown in Table 14.
- Virgin oil versus re-refined oil in lab engine tests.
(Cell C vs Cell D) as shown in Table 15.

- Field service vs lab engine test for a virgin oil.
(Cell A vs Cell C) as shown in Table 16.
- Field service vs lab engine test for a re-refined oil.
(Cell B vs Cell D) as shown in Table 17.

Each deposit comparison table shows the major qualitative deposit differences tabulated by increasing temperature area of the piston (skirt, undercrown, and crown) and by increasing polar solubility of the deposit. A summary of the significant trends and observations of each comparison is presented in the following sections.

TABLE 14. DEPOSIT COMPARISON OF CELL A VS CELL B
(FIELD VIRGIN AND RE-REFINED OILS)

Increasing Temperature →				
	<u>Fraction</u>	<u>Skirt</u>	<u>Undercrown</u>	<u>Crown</u>
	Heptane	Similar	V-more S, N	V-more S, N, higher MW
Increasingly Polar Solvents	Toluene	V-more S, N	V-more S, N	V-more S
	TAM	R-more S	V-more N	V-more S, N
	DMF	V-more S, N	V-more S R-more N	V-more N R-more S
	Filter	R-add. package elements present		

V = Virgin oil
R = Re-refined oil

TABLE 15. DEPOSIT COMPARISON OF CELL C VS CELL D
(LAB VIRGIN AND RE-REFINED OILS)

Increasing Temperature →				
	<u>Fraction</u> Heptane	<u>Skirt</u> R-more N	<u>Undercrown</u> V-more S, N	<u>Crown</u> Similar
Increasingly Polar Solvents	Toluene	V-more S	R-more deposit R-more S	R-more deposit R-more N, S higher MW
	TAM	V-more S	V-more S	V-more S, N
	DMF	R-more deposit V-more S Pb, Br, Cl	V-more S Pb, Br, Cl	V-more S R-higher MW Pb, Br, Cl

V = Virgin oil

R = Re-refined oil

TABLE 16. DEPOSIT COMPARISON OF CELL A VS CELL C
(FIELD VS LAB) VIRGIN OILS

Increasing Temperature →				
	<u>Fraction</u> Heptane	<u>Skirt</u> L-more S, deposit	<u>Undercrown</u> Similar	<u>Crown</u> L-more deposit F-higher MW
Increasingly Polar Solvents	Toluene	F-more N	F-more S	L-more S, N, deposit
	TAM	F-more deposit Higher MW L-more S	F-more S, deposit	L-more Br F-more N, S, Mn deposit
	DMF*	F-more N, deposit L-more S	F-more deposit L-more S	F-more deposit L-more S
	Filter	L-more S, Pb F-more Mn, add. elements	- -	F-more N, add. elements

L = Lab engine test

F = Field engine

*DMF lab fractions all reflect fuel used (contain Pb, Cl, Br)

TABLE 17. DEPOSIT COMPARISON OF CELL B VS CELL D
(FIELD VS LAB) RE-REFINED OILS

Increasing Temperature →				
	<u>Fraction</u>	<u>Skirt</u>	<u>Undercrown</u>	<u>Crown</u>
	Heptane	L-more N, S, deposit	L-more deposit Cl	L-more deposit S, Cl
Increasingly Polar Solvents	Toluene	F-more deposit, S	L-more N, S F-more deposit	L-more N, S higher MW F-more deposit
	TAM	F-more S, deposit	F-more S Both high N	F-more S, N, deposit
	DMF*	F-more S	F-more S, N, deposit	F-more S, N, deposit
	Filter	F-Mn L-Pb	- -	F-Mn L-Pb

L = Lab engine test

F = Field engine

* DMF lab fractions all reflect fuel used (contain Pb, Cl, Br)

C. Discussion

1. Field Service Deposits: Virgin Oil (Cell A) vs Re-refined Oil (Cell B)

In examining the deposits from field service engines, some differences were observed in virgin oil-derived (V) and re-refined-derived (R) deposit composition as shown in Table 14. In the less polar fractions, the V deposit tended to have higher sulfur and usually higher nitrogen content for all piston areas. In the more polar fractions, the R deposit occasionally had more sulfur or nitrogen. For the R deposits, the insoluble material which collected on the filter after washing contained mainly additive-related elements.

2. Laboratory Engine Deposits: Virgin Oil (Cell C) vs Re-refined Oil (Cell D)

Table 15 shows a qualitative comparison of deposits from laboratory engine (Sequence IIID) tests of virgin (V) and re-refined (R) engine oils. In the polar-soluble deposit fractions (DMF and TAM), the V oil deposit consistently had a higher sulfur content than the R oil deposit. While in the toluene fraction from the piston crown, the R oil deposit had higher nitrogen, sulfur, and molecular weight. No other strong compositional trends were observed for this comparison.

3. Virgin Oil Deposits: Field Service (Cell A) vs Lab Engine (Cell C)

Table 16 shows a qualitative comparison of virgin oil deposits from laboratory engine (L) and field service (F) engines. The major differences in field and lab deposit compositions were:

- The F deposits contained more polar-soluble material from the skirt, undercrown, and crown areas.
- The lab DMF-soluble deposits contained more sulfur for each piston area.
- Fuel differences such as lead content were observed in deposit compositions.

4. Re-refined Oil Deposits: Field Service (Cell B) vs Lab Engine (Cell D)

Table 17 shows a qualitative comparison of re-refined oil deposits from laboratory engine (L) and field service (F) engines. The main differences in field and lab deposit compositions were:

- In the polar fractions, the F deposit generally had more sulfur and nitrogen and was present in a greater quantity.
- In most of the nonpolar fractions, the L deposit generally contained more sulfur and nitrogen.

In summary, the deposit analyses revealed that some differences exist in the chemical composition of virgin oil and re-refined oil deposits.

V. SINGLE-CYLINDER ENGINE TESTS

A. Blowby Diversion Tests

The importance of engine blowby composition in the mechanism of engine varnish formation was determined by several researchers.(6-13) The U.S. Army Fuels and Lubricants Research Laboratory has previously developed a blowby sampling apparatus which allows sampling of virgin blowby gases from the piston ring zone (14). The objective of this phase of the program was to utilize the blowby diversion technique to determine if virgin and re-refined oils produced "substantially equivalent" blowby when a common fuel is used. If the oils produced the same blowby with a common fuel, this would be another indication of "substantial equivalency" between virgin and re-refined oils.

The single-cylinder Coordinated Lubricants Research (CLR) engine described in Table 18 was fitted with a blowby diversion piston as illustrated in Figure 4. The engine was operated at conditions which simulate Cycle II of the Sequence VD test, as shown in Table 19. Engine blowby was collected and analyzed to determine re-refined and virgin oil equivalency. A schematic of the system used for the collection of blowby from the engine ring belt area is shown in Figure 5. Blowby was passed through 0°C ice water and dry ice/isopropyl alcohol cold traps with the remaining gaseous blowby trapped in a Tedlar bag. The bag samples were analyzed for NO, NO_x, UBHC, CO, CO₂, and O₂ using standard exhaust emission techniques. A second blowby collection/analysis system (Figure 6) was used to determine the carbonyl compounds

**TABLE 18. COORDINATED LUBRICANTS RESEARCH (CLR)
ENGINE CHARACTERISTICS**

Displacement	42.5 in. ³
Bore and Stroke	3.80 in. X 3.75 in.
Compression Ratio	8.3:1
Piston	Aluminum, 3-Ring
Piston Rings	Barrel-Faced Chrome; 1st Comp., Taper Face Cast Iron; 2nd Comp., Two Chrome Rails and Expander, Oil Control
Cylinder	Replaceable Cast Iron Sleeve
Oil Capacity	1 Quart (no filter)

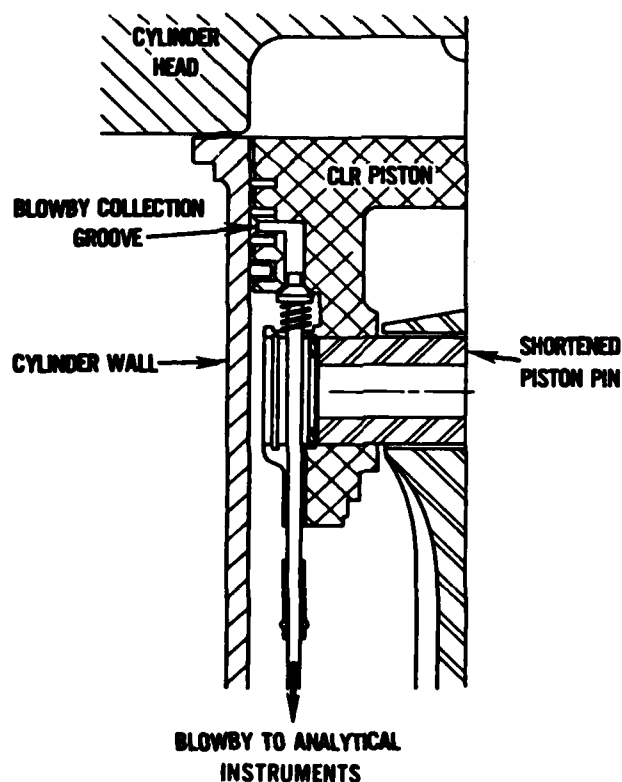


FIGURE 4. BLOWBY DIVERSION PISTON

TABLE 19. OPERATING CONDITIONS FOR
BLOWBY DIVERSION COLLECTION
CLR ENGINE

RPM	2500
Torque N·M (lb-ft)	35.2 (26)
Air/Fuel Ratio	15.6
Coolant Out Temp, °C (°F)	77 (170)
Oil Gallery Temp, °C (°F)	93 (200)
Exhaust Temp, °C (°F)	699 (1290)
Spark Timing, °BTDC	26

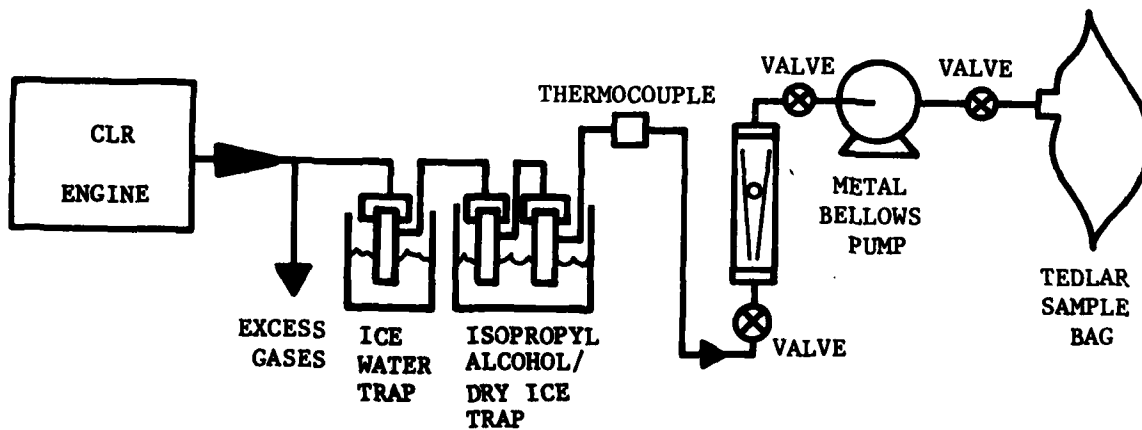


FIGURE 5. SYSTEM FOR COLLECTION OF BLOWBY GAS SAMPLES FOR ANALYSIS

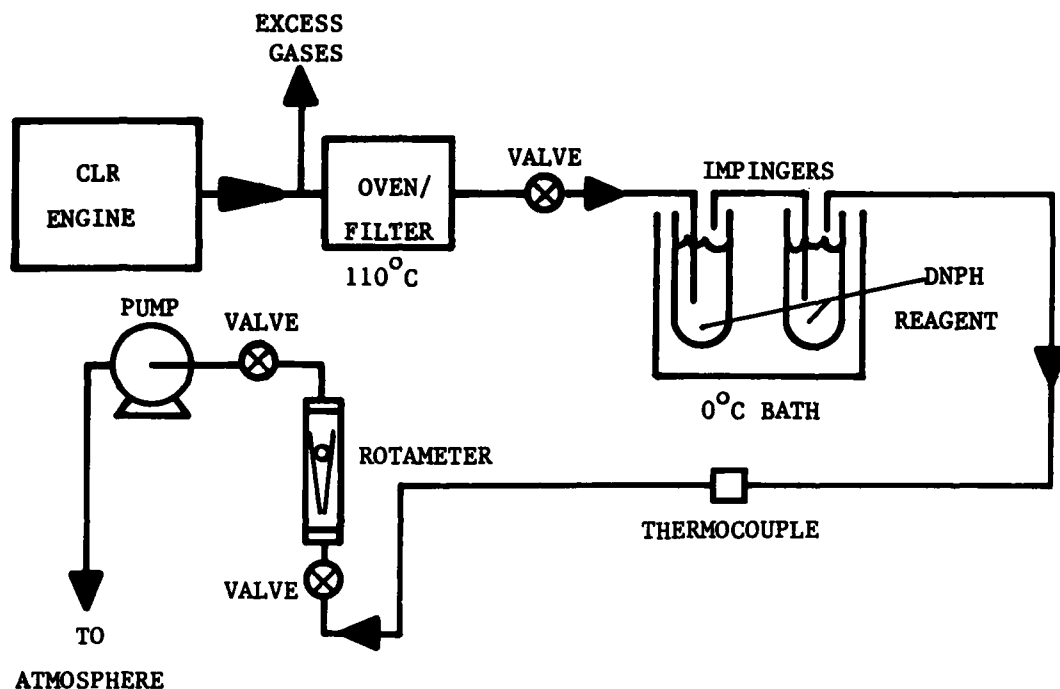


FIGURE 6. SYSTEM FOR COLLECTION OF BLOWBY GAS SAMPLES FOR CARBONYL COMPOUND ANALYSIS

(aldehydes and ketones) present in the blowby.⁽¹⁵⁾ This technique consisted of bubbling the blowby through glass impingers containing 2, 4-dinitrophenylhydrazine (DNPH) in dilute hydrochloric acid. The carbonyl compounds react with the DNPH to form phenylhydrazone derivatives. The derivatives are recovered and identified using a gas chromatographic procedure.

The blowby test matrix consisted of two fuels (Phillips J unleaded gasoline and isooctane) and two lubricant basestocks (one virgin and one re-refined). The virgin basestock (AL-10575) was a solvent neutral oil from a Port Arthur, TX refinery, while the re-refined basestock (AL-8481) came from an Oklahoma re-refiner. General inspection properties of both AL-10575 and AL-8481 are presented in Table 20.

TABLE 20. TEST LUBRICANT PROPERTIES

<u>Property</u>	<u>Test Method</u>	<u>Virgin Basestock AL-10575</u>	<u>Re-refined Basestock AL-8481</u>
KVis, 40°C, cSt	D 445	62.21	56.61
KVis, 100°C, cSt	D 445	8.22	8.04
Viscosity Index	D 2270	100	109
Total Acid No.	D 664	0.01	0.01
API Gravity, °	D 287	29.2	30.0
Flash point, °C	D 92	237	192
Sulfur, wt%	D 2622	0.39	0.16
Carbon Residue, wt%	D 524	0.07	0.13
Pour point, °C	D 97	-12	-10

The results of the blowby gas analyses are presented in Table 21. Blowby was collected by the methods shown in Figures 5 and 6. No substantial differences in blowby composition were detected for any of the lubricant/fuel combinations.

TABLE 21. ANALYSIS OF BLOWBY (BAG SAMPLES)

Basestock:	Re-refined	Virgin	Re-refined	Virgin
Code:	AL-8481	AL-10575	AL-8481	AL-10575
Fuel:	<u>Phillips J</u>	<u>Phillips J</u>	<u>Isooctane</u>	<u>Isooctane</u>
<u>Gas Analyses</u>				
NO, ppm	73	69	68	71
NO _x , ppm	102	92	91	95
UBHC, ppm C	18,770	18,380	18,720	18,500
CO, %	0.019	0.025	0.028	0.028
CO ₂ , %	1.15	1.20	1.12	1.15
O ₂ , %	19.4	19.4	19.3	19.3
<u>Carbonyl Compounds, ppm</u>				
Aldehydes	3	8	13	4
Ketones	<1	2	<1	<1

The materials collected in the cold traps were analyzed by gas chromatography and infrared spectroscopy. IR traces are presented for isooctane (Figure 7) and Phillips J (Figure 8) fuels. The 0°C trap material for each fuel/lubricant combination had a very similar IR trace and a representative trace is shown in Figure 9. The gas chromatograms of the 0°C trap material were very similar for a given fuel regardless of the lubricant used. Figure 10 is a typical chromatogram for 0°C trap material when using Phillips J unleaded gasoline. Figure 11 is a typical chromatogram of the 0°C trap material when using isooctane. Figure 12 is a chromatogram of pure isooctane which, when compared with Figure 11, shows that isooctane (fuel) was the primary material collected in the 0°C trap. In examining the material collected in the -40°C traps, it was found that both IR traces and gas chromatograms were similar for a given fuel regardless of the lubricant used. The tests which used isooctane as the fuel contained primarily isooctane in the -40°C trap. Overall, it can be concluded that the fuel was the primary determinant of the blowby composition. The virgin and re-refined lubricants were equivalent in that both contributed very little, if any, to the blowby.

B. CLR Engine Deposit Tests

Several researchers have reported on the effects of a number of variables on engine varnish and sludge.(6-13) Among variables investigated were: fuel composition effects, nitrogen oxide's role in engine deposit formation, engine characteristics (compression ratio, jacket temperature) and engine operating conditions (speed, load, temperature). Little information was found concerning the role of re-refined base oils in engine deposits formation. Spilners, et al., recently reported that a re-refined base oil produced better varnish and sludge merit ratings than a solvent-refined neutral oil.(16) Thus, a series of engine tests were conducted using the single-cylinder CLR gasoline engine to determine and compare the deposit-forming tendencies of virgin and re-refined basestocks. This study investigated base oil equivalence from a deposit formation tendency.

A test cycle was chosen which was similar to the procedure developed and used by Spilners, except that no additional NO_x was injected into the engine, and

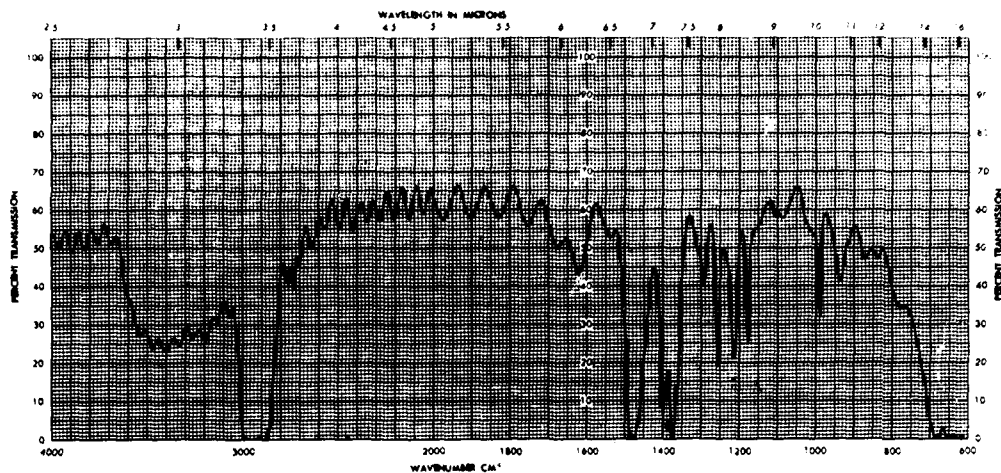


FIGURE 7. INFRARED SPECTRA FOR FUEL: ISOOCTANE

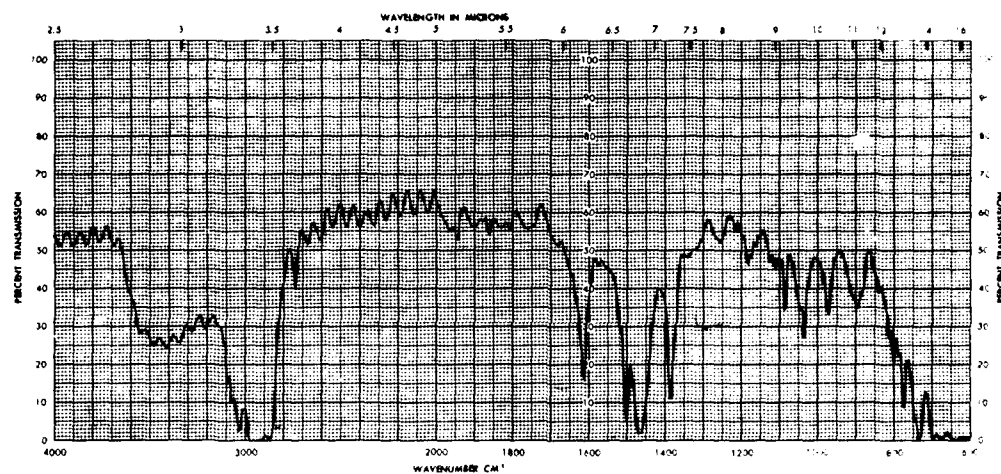


FIGURE 8. INFRARED SPECTRA FOR FUEL: PHILLIPS J

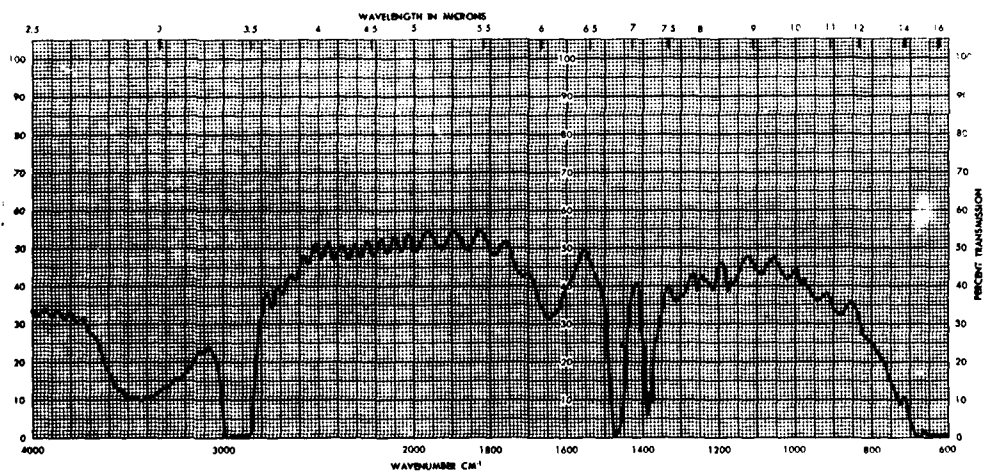


FIGURE 9. INFRARED SPECTRA FOR 0°C TRAP MATERIAL (TYPICAL)

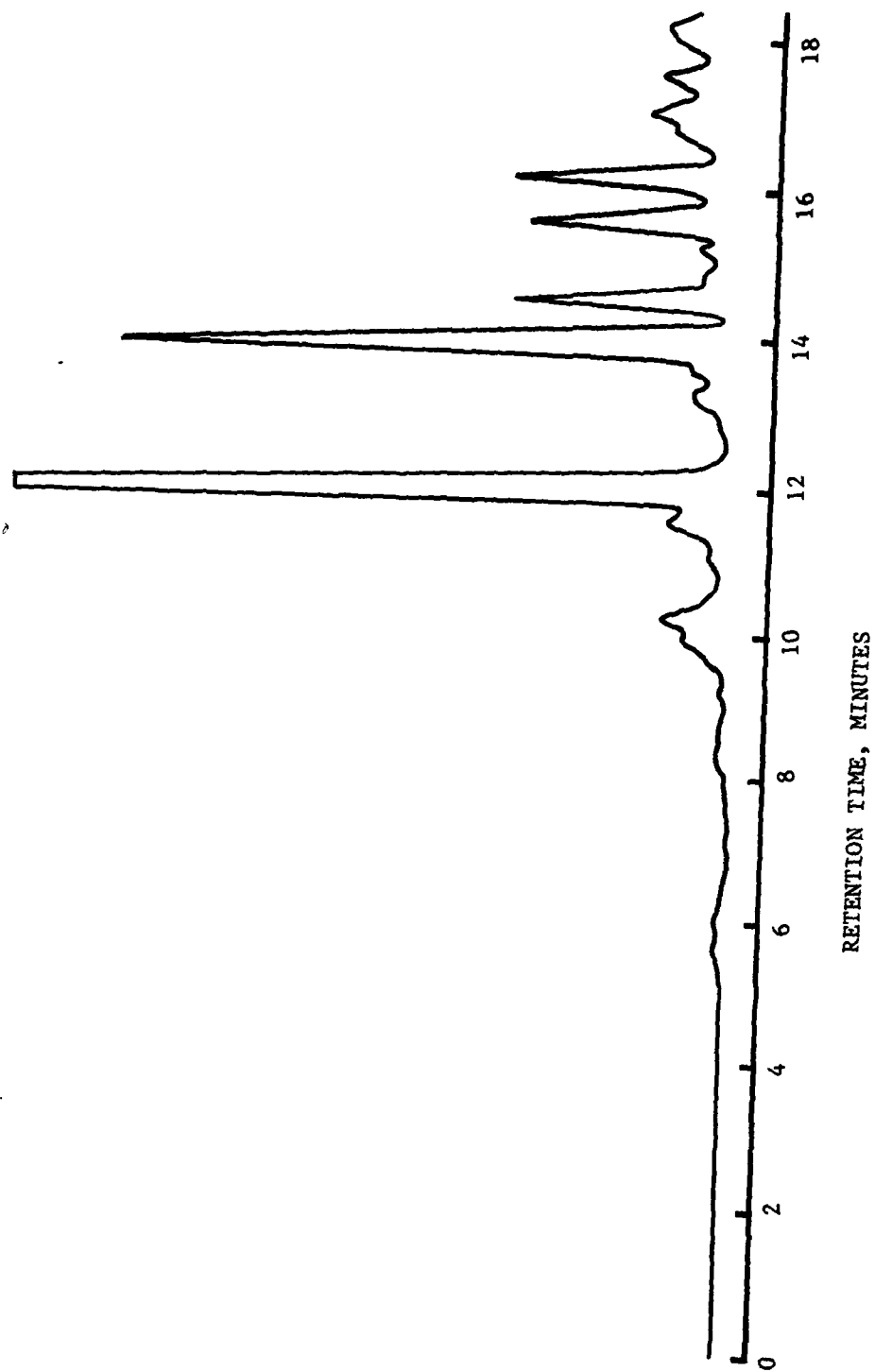


FIGURE 10. GAS CHROMATOGRAM OF 0°C TRAP MATERIAL WHEN USING PHILLIPS J GASOLINE

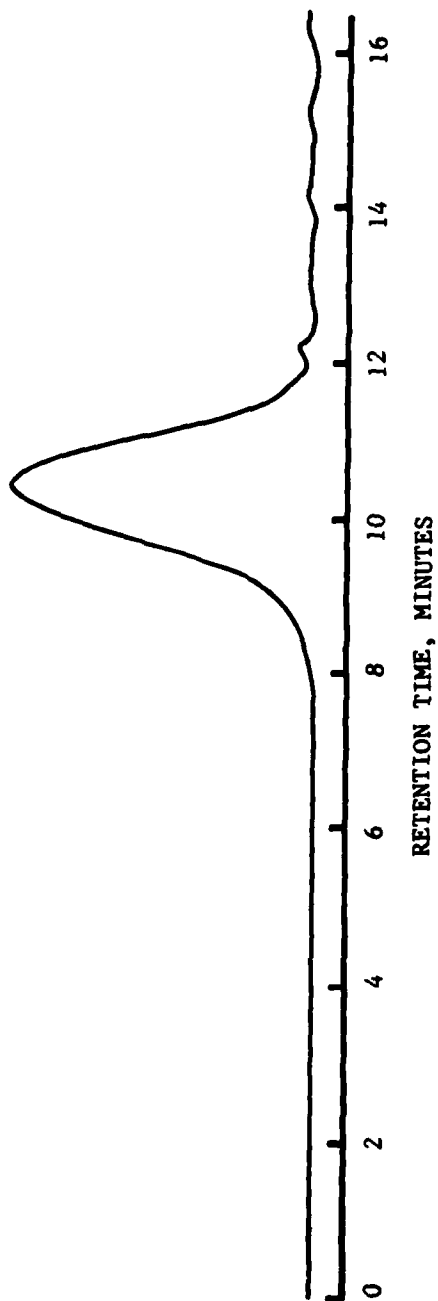


FIGURE 11. GAS CHROMATOGRAM OF 0°C TRAP MATERIAL WHEN USING ISOCTANE AS FUEL

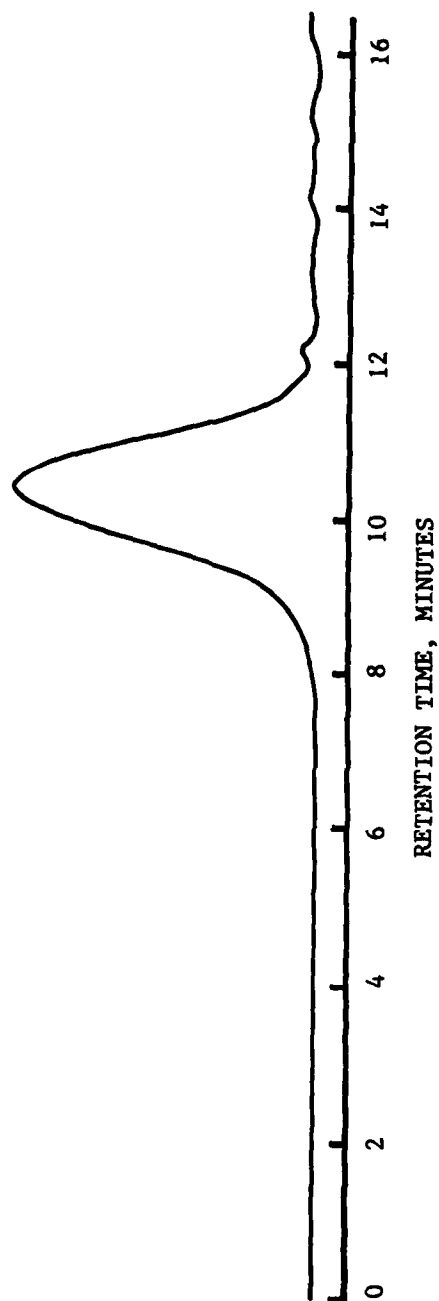


FIGURE 12. GAS CHROMATOGRAM OF ISOCTANE

the test time was increased to 100 hours.(17) The CLR engine deposit test operating conditions are shown in Table 22. Fuel additions were made to the crankcase every 12 hours to increase test severity. The fuel used for all the CLR deposit tests was Phillips J unleaded reference gasoline. The properties of Phillips J test fuel are shown in Table 23.

TABLE 22. CLR DEPOSIT TEST CONDITIONS

Test hours	100
Speed, rpm	2000±25
Load, lb-ft (N-m)	30±1 (40.7±1.4)
Spark advance, deg	20±1
Air/Fuel ratio	15.75±0.25:1
Oil gallery temp, °F (°C)	260±2 (127)
Coolant out temp, °F (°C)	190±2 (88)
Blowby rate, ft ³ /hr, (m ³ /hr)	36±2 (1.02)
Fuel additions to crankcase, cm ³	100 every 12 hours

The CLR deposit test matrix consisted of two virgin-base oils and two re-refined base oils which were each tested using Phillips J unleaded gasoline. Re-refined base oil A (AL-8481) was received from a California supplier. An earlier batch of this oil was formulated with appropriate additives and had previously passed all the engine performance tests of MIL-L-46152 (API service SE-CC) when tested during the joint EPA/DOD re-refined engine oil program.(18) Re-refined base oil B (AL-8181) was supplied by an Oklahoma re-refiner. Virgin base oil C (AL-10575) was a solvent-refined neutral oil obtained from a Port Arthur, TX refinery, while virgin base oil D (AL-10576) was a light-intermediate, solvent neutral oil obtained from a Louisiana refinery. The inspection properties for these four base oils are presented in Table 24. The re-refined base oils had darker color, and higher saponification numbers than the virgin base oils. Also re-refined oil B had a TAN of 0.35 which is higher than normal for a base oil. The re-refined base

oils were analyzed for residual additive and wear element content as shown in Table 25. Based on these analyses, both re-refined oils were free of residual contaminant and additive content. Neither the virgin nor re-refined base oils contained any additive treatment.

TABLE 23. PROPERTIES OF PHILLIPS J UNLEADED GASOLINE

<u>Property</u>		
Gravity, API, 15.6°C	D 287	52.8
Gravity, Specific, 15.6°C	D 1298	0.7678
Copper Corrosion, 3 hr @ ₂ 100°C	D 130	1A
Reid Vapor Pressure, kg/m ²	D 323	43.9
Octane Number, Research	D 2699	95.9
Octane Number, Motor	D 2700	85.0
(R+M)/2		90.5
Total Sulfur, wt%	D 1266	0.020
Gum, mg/100 ml	D 381	0.6
Oxidation Stability, min	D 525	1200+
Distillation, % Evap., °C	D 86	
IBP		32
5%		41
10%		48
15%		54
20%		60
30%		77
40%		97
50%		114
60%		119
70%		127
80%		139
90%		162
95%		181
E.P.		210
Recovery, %		98.5
Residue, %		0.5
Hydrocarbon Types, vol%	D 1319	
Saturates,		42
Olefins		12
Aromatics		46

TABLE 24. BASE OIL INSPECTION PROPERTIES

Property	Test Method	Test Oil			
		A AL-8481	B AL-8181	C AL-10575	D AL-10576
KVis, 40°C, cSt	D 445	56.61	68.06	62.21	66.31
KVis, 100°C, cSt	D 445	8.04	8.99	8.22	8.41
Viscosity Index	D 227	109	106	100	96
Total Acid No.	D 664	0.01	0.35	0.01	0.02
API Gravity, °	D 287	30.0	28.8	29.2	29.8
Flash Point, °C	D 92	192	215	237	245
Pour Point, °C	D 97	-10	-21	-12	-12
Carbon Residue, wt%	D 524	0.13	0.15	0.07	0.06
Sulfur, wt%	D 2622	0.16	0.20	0.39	0.13
Saponification No.	D 94	0.77	1.57	0.05	0.10
Color	D 1500	5.0	5.5	2.0	1.5
Aromaticity	UV				
Wt% ring carbon					
Mono-ring		3.8	3.5	3.9	2.2
Di-ring		1.7	1.6	1.2	0.7
Tri-ring		0.3	0.2	0.1	0.1

UV = Ultraviolet spectroscopy method

TABLE 25. ADDITIONAL RE-REFINED BASE OIL INSPECTIONS

Elemental Content, ppm by AA	Oil A AL-8481	Oil B AL-8181
Calcium	5	< 5
Barium	< 5	< 5
Zinc	< 2	< 2
Lead	1	1
Magnesium	< 1	< 1
Copper	< 5	< 5
Chromium	< 5	< 5
Iron	< 2	< 2
Sodium	< 2	< 2
Phosphorus (by Modif. Oronite Method)	< 10	< 10

The results of the four CLR engine deposit tests are shown in Table 26. The two re-refined base oils (A and B) tended to have more varnish in lower temperature nonrubbing oil-wetted areas such as the push rod cover and rocker arm cover. In the hotter cylinder wall area, the virgin base oils (C and D) had more varnish. Overall, average engine varnish was about the same for each oil, except for virgin Oil D, which had less average varnish. Average engine sludge ratings were about the same for all four base oils. Virgin Oil D performance was somewhat unusual in that oil screening plugging was rather high (75 percent), yet its overall engine varnish and sludge merit ratings were the highest. The used oil analyses (Table 26) showed a slight increase in TAN, viscosity, and insolubles for Oils A, C and D, with a slightly higher increase in these properties for Oil B. Iron and copper wear metal accumulations were low for all four base oils.

Differential infrared analysis was conducted on the used oils from the CLR engine deposit tests. Each set of a new and a used oil from a CLR test was run in the same cell to ensure that the path length did not vary. Sodium chloride windows were used. New and used oils AL-8481-L and AL-10575-L were scanned with a pathlength of 0.024 mm, oils AL-10576-L and AL-8181-L with a pathlength of 0.05 mm. A Microlab 620 MX computing infrared spectrophotometer with a spectral manipulation Compuset microprocessor module was used to subtract each new oil spectrum from the corresponding used oil spectrum.

The resulting spectra (Appendix B) show the products formed in the used oils. Absorbance bands near 1710 cm^{-1} represent carboxyl groups (C=O) due to oxidation of the new oil. Bands near 1640 cm^{-1} represent nitro groups ($-\text{O}-\text{NO}_2$) due to nitration of the new oil. Bands near 1550 cm^{-1} represent carboxylate salts ($\text{O}=\text{C}-\text{O}^-$) and nitro groups ($-\text{C}-\text{NO}_2$) due to nitration and oxidation. Bands near 1280 cm^{-1} represent nitro groups ($-\text{O}-\text{NO}_2$) and carbon-oxygen bonds ($\text{C}-\text{O}$) due to nitration and oxidation of the new oil. Bands near 870 cm^{-1} represent nitrogen-oxygen bonds ($\text{N}-\text{O}$) due to nitration of the new oil. Absorbance of bands at 1710 , 1640 , 1280 , and 870 cm^{-1} were corrected to the same pathlength and listed in Table 27.

The used re-refined base oils, AL-8181 and AL-8481, appear to have been oxidized and nitrated slightly more than the used virgin-base oils.

TABLE 26. CLR ENGINE DEPOSIT TESTS

Test Method	Re-refined				Virgin			
	Oil A		Oil B		Oil C		Oil D	
	New	Used	New	Used	New	Used	New	Used
Oil Properties								
K. Vis, 40°C, cSt	56.61	64.93	68.06	90.20	62.21	65.43	66.31	66.74
K. Vis, 100°C, cSt	8.04	8.66	8.99	10.69	8.22	8.25	8.41	8.59
TAN	0.01	1.18	0.35	2.32	0.01	0.91	0.02	1.06
Insolubles, wt%								
Pentane A	Nil	0.46	Nil	1.31	Nil	0.26	Nil	0.51
Toluene A	Nil	0.06	Nil	0.18	Nil	0.04	Nil	0.13
Pentane B	Nil	0.18	Nil	0.88	Nil	0.11	Nil	0.35
Toluene B	Nil	0.05	Nil	0.19	Nil	0.04	Nil	0.14
Wear Metals, ppm								
Fe	<10	18	<10	39	<10	22	<10	37
Cu	<10	15	<10	12	<10	13	<10	<10
Pb	<50	<50	<50	<50	<50	<50	<50	<50
XRF								
Engine Deposits								
(10 = clean)								
Varnish								
Rocker Arm Cover	5.2		4.0		6.1		7.2	
Push Rod Cover	3.2		3.4		7.2		8.0	
Cylinder Wall	6.3		4.0		3.2		3.6	
(below Ring Travel)								
Crankcase Cover Plate	4.8		3.8		4.7		5.8	
Piston Skirts	4.6		5.3		3.4		5.1	
Avg Engine Varnish	4.8		4.1		4.9		5.9	
Avg Engine Sludge	9.3		9.2		9.2		9.4	
Oil Screen Plugging, %	35		50		40		75	
LUBTOT @ 246°C (0 = clean)	30		15		30		22	

Re-refined oil B, which shows the most oxidation by this IR method, also had a rather large differential viscosity increase and the largest TAN increase, which are also indicative of oxidation.

The base oils tested in the CLR deposit test were evaluated in the LUBTOT apparatus. The LUBTOT results have been found to be indicative of high-temperature engine deposit levels.(19) Figure 13 shows the relationship of CLR piston varnish merit ratings (y) and the LUBTOT rating (x). As piston

TABLE 27. DIFFERENTIAL IR ANALYSES OF USED OILS

Oil Code	Peak Absorbance at Indicated Frequency			
	1710 cm^{-1}	1640 cm^{-1}	1280 cm^{-1}	870 cm^{-1}
A (re-refined)	0.105	0.172	0.122	0.049
B (re-refined)	0.149	0.185	0.146	0.059
C (virgin)	0.083	0.099	0.067	0.027
D (virgin)	0.075	0.067	0.057	0.025

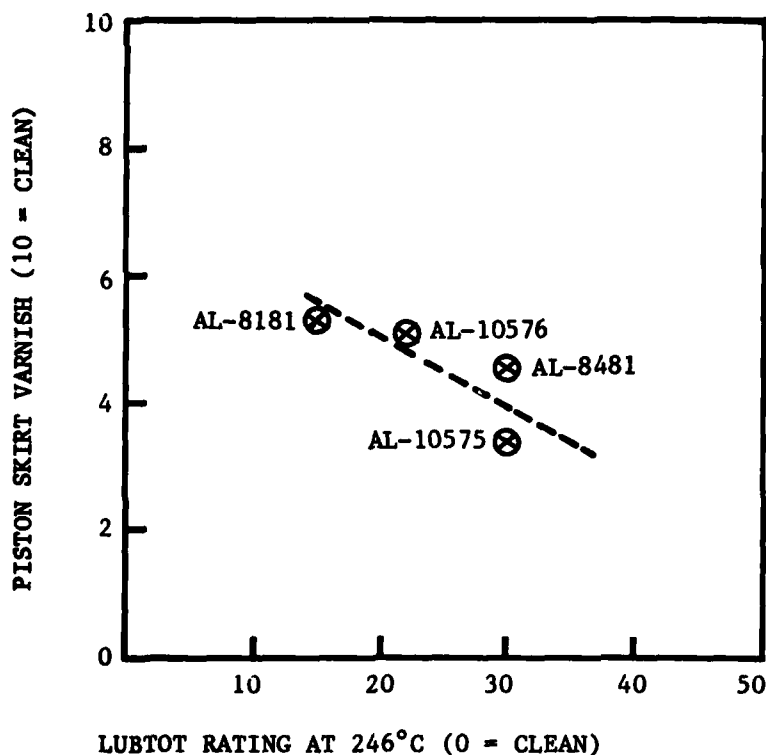


FIGURE 13. RELATIONSHIP OF PISTON VARNISH AND LUBTOT RATING

varnish increased, the LUBTOT rating indicated more deposit was present. This relationship had a correlation coefficient of $R^2 = 0.78$. The average engine varnish and varnish ratings from other engine parts did not appear to correlate with the LUBTOT ratings.

Within the limited scope of this investigation, the only substantial differences observed between virgin and re-refined base oils were: (1) re-refined base oils tended to oxidize and nitrate slightly more than virgin base oils, and (2) re-refined base oils had more varnish in lower temperature nonrubbing areas and less varnish in the hotter cylinder wall area.

VI. ASTM/NBS BASESTOCK CONSISTENCY PROGRAM

A lack of technical information has existed concerning the consistency of both virgin and re-refined basestocks. (20, 21) This issue was addressed by a cooperative ASTM/NBS study of basestock consistency. One of the objectives of the program was to examine the "substantial equivalence" of virgin and re-refined oils from a basestock property consistency standpoint. During the 1-year program, monthly samples of basestock were analyzed by the twelve participating laboratories. Four virgin oils, five re-refined oils, and one control sample were included in the program. (22) MERADCOM/AFLRL participation included the standard property inspection tests and other determinations listed in Table 28. The results of the monthly sample analyses are presented in Appendix C, along with the mean, standard deviation, and high and low values for each oil.

The following preliminary overall conclusions have been made by the task force members: (23)

- Generally, both the virgin oils and the re-refined oils are consistent in production as measured by the tests employed.
- Instances of inconsistency are almost exclusively limited to re-refined oils. However, these inconsistencies are in properties measured and any relationship to basestock quality is yet to be established.

TABLE 28. ASTM/NBS BASESTOCK CONSISTENCY PARTICIPATION

<u>Test</u>	<u>Method</u>
Viscosity	D 445
at 100°C, cSt	
at 40°C, cSt	
Viscosity Index	D 2270
Gravity, °API	D 287
Pour Point, °C	D 97
Carbon Residue, %	D 524
Sulfated Ash, %	D 874
Total Acid Number	D 664
Saponification Number	D 94
Elemental Content, Mass %	
Nitrogen	Chemiluminescent
Chlorine	X-ray Fluorescence*
Sulfur	D 2622**
Color	D 1500
Boiling Point Distribution,	
°C at 1, 5, 10, 50, and	
90% point	D 2877*** (modified)
LUBTOT	AFLRL
UV Aromaticity	AFLRL

* Procedure similar to sulfur method with detection limit of 100 ppm.

** Modified to use energy-dispersive x-ray fluorescence instead of wave length dispersive XRF.

*** Modified for determination of higher boiling components. Data processing on HP 3354 Lab Data System using HP and SwRI software.

The following preliminary conclusions about basestock consistency have been made, based on tests performed by MERADCOM/AFLRL:

Viscosity - Standard deviation of the re-refined oils was equivalent to the virgin oils for three companies and higher for two.

Sulfur, Gravity and Carbon Residue - Equivalent consistency.

Pour Point and VI - Equivalent with exception of three re-refined oils.

Color and Sulfated Ash - Equivalent with exception of three re-refined oils.

Sap No. - All five re-refined oils had higher values and greater standard deviations.

Nitrogen - Four re-refined oils had higher variations.

After all the data from each participating laboratory are compiled and statistical analyses performed, the ASTM/NBS Basestock Consistency Task Force will make definitive conclusions.

VII. CONCLUSIONS

The following conclusions are drawn from this work:

- Deposit analysis investigations revealed that some differences exist in the chemical composition of engine deposits derived from virgin and re-refined engine oils. Primary areas of deposit differences were:

1. Amount of deposit
2. Polar solubility of deposit
3. Nitrogen and sulfur content of deposit

While deposit composition differences exist, the data did not contain any strong overall trends which would consistently differentiate re-refined and virgin oils.

- Blowby diversion investigations revealed that fuel was the primary determinant of blowby composition. Virgin and re-refined base oils were equivalent in that blowby composition was independent of base oil type.
- In single-cylinder CLR deposit tests, the following substantial differences were observed:
 1. Re-refined base oils tended to be oxidized and nitrated slightly more than virgin base oils.
 2. Re-refined base oils had more varnish in lower temperature nonrubbing areas and less varnish in the hotter cylinder wall area.
- Generally, both virgin oils and re-refined oils are consistent in production as measured by the tests used.
- Instances of inconsistency are almost exclusively limited to re-refined oils. Final conclusions with respect to basestock consistency will be made by the ASTM/NBS Basestock Consistency Task Force.

VIII. LIST OF REFERENCES

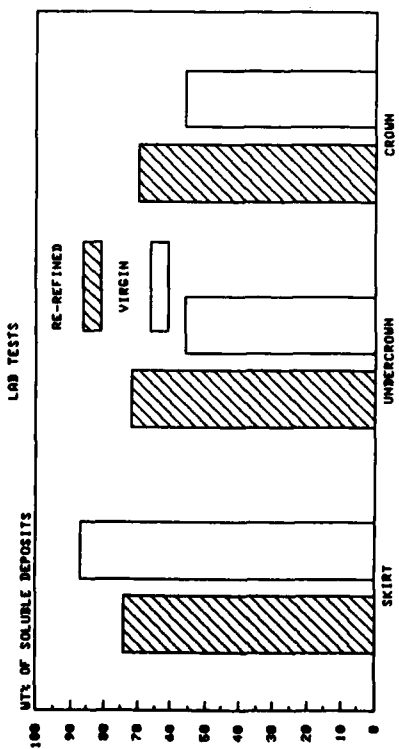
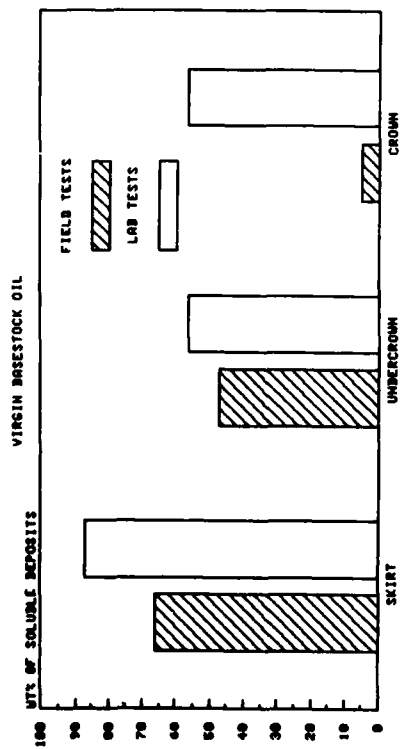
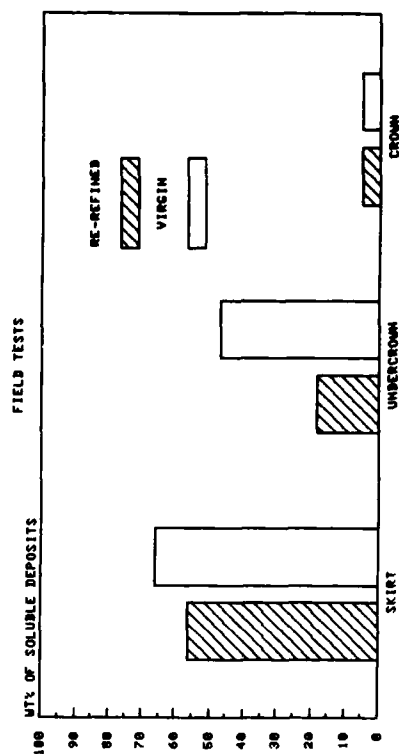
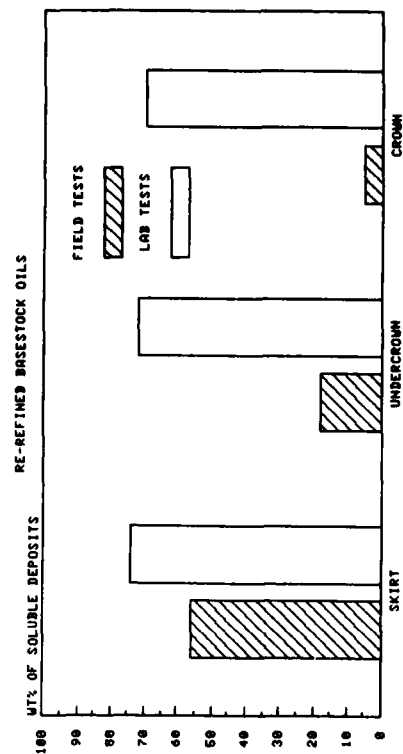
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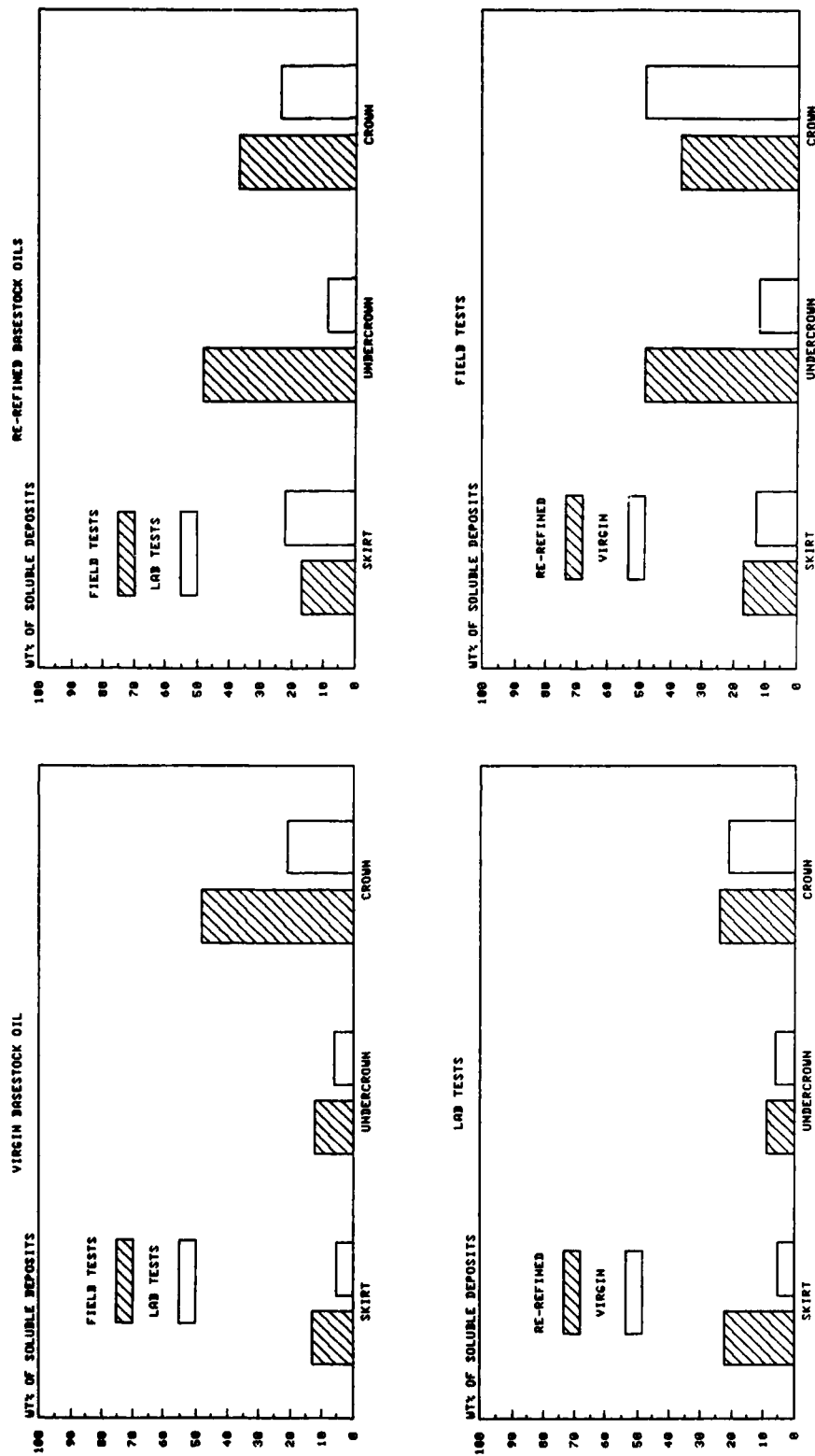
APPENDIX A

BAR CHARTS OF DEPOSIT DISTRIBUTIONS

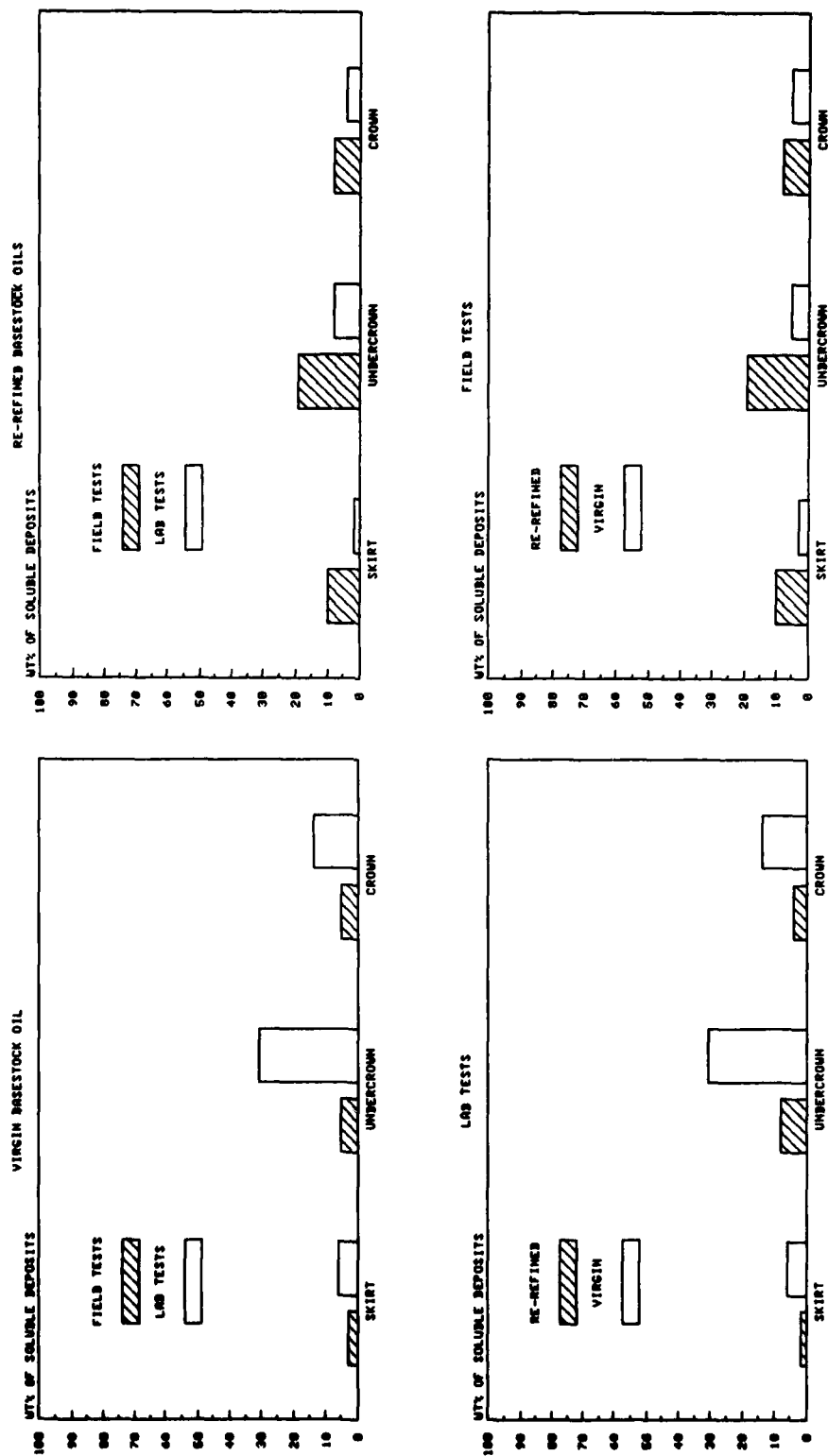
DISTRIBUTION OF HEPTANE SOLUBLE DEPOSITS



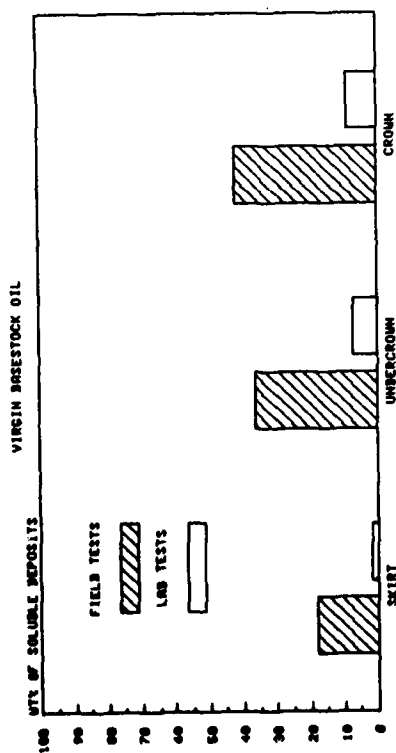
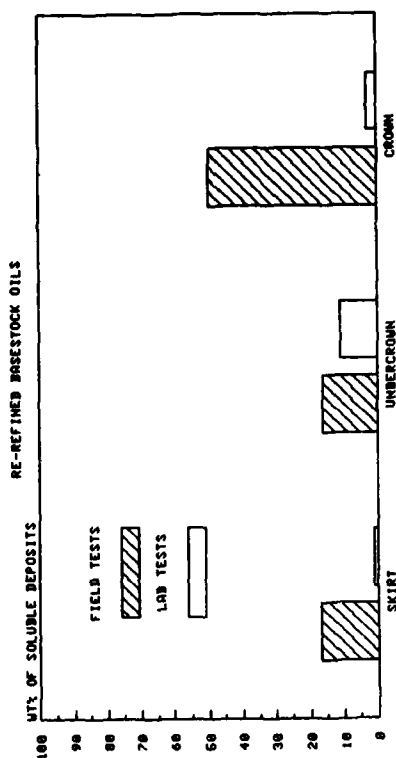
DISTRIBUTION OF DMF SOLUBLE DEPOSITS



DISTRIBUTION OF TOLUENE SOLUBLE DEPOSITS

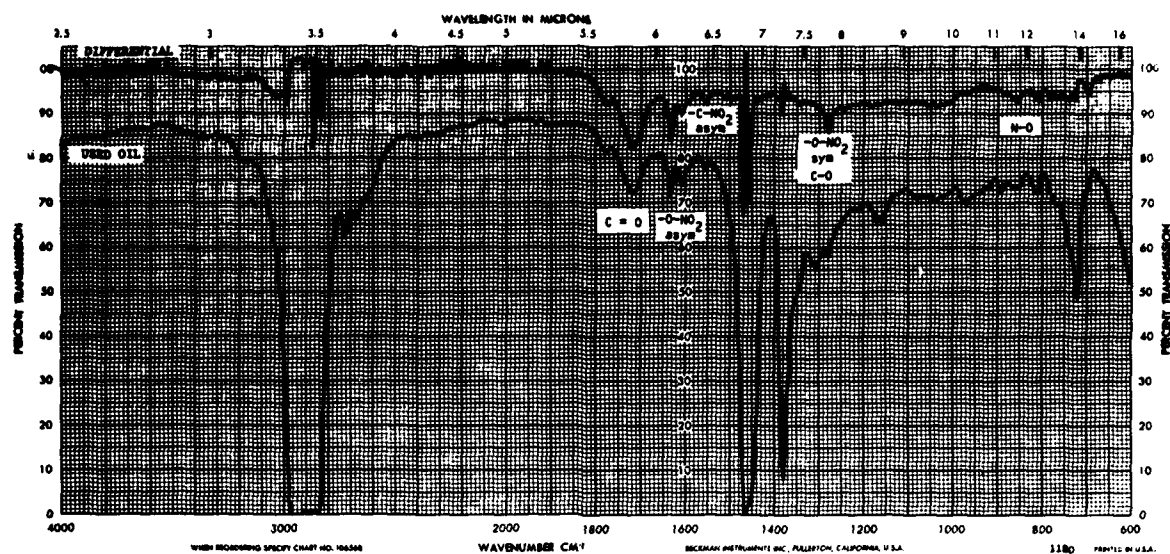


DISTRIBUTION OF TAN SOLUBLE DEPOSITS

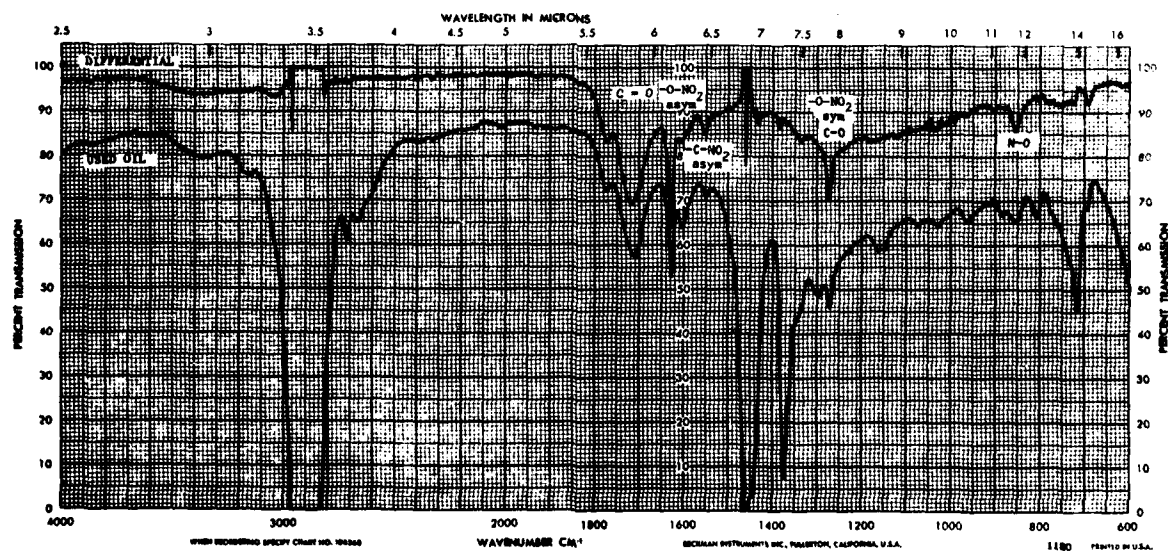


APPENDIX B

USED OIL INFRARED SPECTRA



INFRARED SPECTRA FOR CLR TEST: AL-10576-L



INFRARED SPECTRA FOR CLR TEST: AL-8181-L

APPENDIX C

**TABULATED DATA FROM ASTM-NBS
BASESTOCK CONSISTENCY
STUDY**

KINEMATIC VISCOSITY AT 40° C, cSt (D 445)

Date	Virgin Stocks				Reference I	Re-refined Stocks				
	A	B	C	D		E	F	G	H	J
Mar 80	-	-	-	-	38.93	-	-	-	-	-
Mar 80	23.39	-	31.08	48.66	38.87	26.59	62.11	28.82	66.25	37.61
Apr 80	24.61	53.16	29.98	48.16	38.75	25.46	57.88	28.47	64.50	42.79
May 80	24.08	56.43	30.80	47.00	38.94	24.60	56.69	28.95	61.39	41.11
Jun 80	23.97	56.87	32.00	46.69	38.93	26.57	57.74	29.79	62.40	39.88
July 80	24.76	57.28	30.25	49.13	38.98	25.42	59.48	29.80	55.43	42.84
Aug 80	23.87	58.53	30.58	48.48	38.67	25.13	57.55	32.93	64.07	41.31
Sep 80	23.39	55.93	31.34	49.53	38.71	28.19	58.29	30.68	57.67	41.49
Oct 80	23.57	59.27	30.58	50.14	38.84	30.72	62.02	30.65	68.32	38.60
Nov 80	23.91	53.88	29.31	49.02	38.92	28.09	61.14	28.64	58.81	46.91
Dec 80	23.27	58.99	29.63	49.51	38.88	31.03	61.80	29.72	58.02	49.52
Jan 81	24.66	58.26	32.09	50.07	38.74	-	57.38	27.01	62.23	42.92
Feb 81	24.94	63.00	30.05	49.23	38.76	-	-	31.07	57.26	-
Mean	24.04	57.42	30.64	48.80	38.84	27.18	59.28	29.71	61.03	42.27
Std. Dev	0.58	2.70	0.87	1.09	0.10	2.28	2.10	1.52	3.90	3.45
High	24.94	63.00	32.09	50.14	38.98	31.03	62.11	32.93	68.32	49.52
Low	23.27	53.16	29.31	46.69	38.71	24.60	56.69	27.01	55.43	37.61

KINEMATIC VISCOSITY AT 100°C, cSt (D 445)

Date	Virgin Stocks				Reference I	Re-refined Stocks				
	A	B	C	D		E	F	G	H	J
Mar 80	-	-	-	-	6.10	-	-	-	-	-
Mar 80	4.52	-	5.40	6.89	6.10	4.76	8.64	4.99	8.82	5.83
Apr 80	4.61	7.48	5.25	6.83	6.11	4.64	8.25	4.92	8.69	6.34
May 80	4.56	7.77	5.34	6.81	6.14	4.55	8.17	4.98	8.48	6.19
Jun 80	4.55	7.77	5.46	6.74	6.10	4.77	8.31	5.01	8.64	6.06
July 80	4.60	7.80	5.23	6.92	6.10	4.63	8.35	5.09	7.93	6.41
Aug 80	4.52	7.93	5.27	6.93	6.11	4.92	8.15	5.58	8.83	6.27
Sep 80	4.47	7.68	5.33	7.00	6.12	4.97	8.32	5.22	8.33	6.26
Oct 80	4.49	8.00	5.25	7.05	6.11	5.59	8.68	5.29	9.20	5.95
Nov 80	4.52	7.47	5.18	6.97	6.13	4.93	9.01	4.97	8.31	6.78
Dec 80	4.44	7.97	5.16	7.01	6.11	5.23	8.68	5.08	8.24	7.00
Jan 81	4.61	7.91	5.40	7.07	6.12	-	8.55	4.77	8.66	6.36
Feb 81	4.62	8.35	5.21	6.99	6.11	-	-	5.27	8.20	-
Mean	4.54	7.83	5.29	6.93	6.11	4.89	8.46	5.10	8.53	6.31
Std. Dev	0.06	0.25	0.10	0.10	0.01	0.32	0.27	0.21	0.35	0.34
High	4.62	8.35	5.46	7.07	6.13	5.59	9.01	5.58	9.20	7.00
Low	4.44	7.47	5.16	6.74	6.10	4.55	8.15	4.92	7.93	5.83

VISCOSITY INDEX (D 2270)

Date	Virgin Stocks				Reference I	Re-refined Stocks				
	A	B	C	D		E	F	G	H	J
Mar 80	-	-	-	-	101	-	-	-	-	-
Mar 80	101	-	108	95	101	96	112	97	106	94
Apr 80	102	102	106	95	102	96	112	93	107	94
May 80	102	102	106	98	103	96	113	94	109	95
Jun 80	103	100	106	97	101	97	114	90	111	95
July 80	100	100	103	95	101	96	111	96	109	97
Aug 80	101	101	103	98	103	121	110	107	112	98
Sep 80	101	100	102	97	103	100	113	100	115	97
Oct 80	101	101	102	97	102	122	113	104	111	95
Nov 80	101	99	106	97	103	98	124	96	111	97
Dec 80	100	101	103	97	102	98	114	96	111	97
Jan 81	101	101	102	97	103	-	122	93	112	95
Feb 81	99	101	103	97	102	-	-	100	112	-
Mean	101	100.7	104.2	96.7	102.1	102	114.4	97.2	110.5	95.8
Std. Dev	1.0	0.9	2.1	1.1	0.9	10.4	4.5	4.9	2.4	1.4
High	103	102	108	98	103	122	124	107	115	98
Low	99	99	102	95	101	96	110	90	106	94

GRAVITY, °API (D 287)

Date	A	Virgin B	Stocks C	D	Reference I	E	Re-refined Stocks F G	H	J
Mar 80	-	-	-	-	29.9	-	-	-	-
Mar 80	33.0	-	31.2	30.8	29.9	30.5	29.8	29.6	29.2
Apr 80	32.9	30.2	31.1	30.8	29.9	30.6	29.8	29.2	28.8
May 80	33.0	29.9	31.0	30.9	29.8	30.7	29.9	29.4	29.0
Jun 80	33.0	30.2	30.7	30.8	29.9	30.6	29.5	28.4	29.7
July 80	29.9	30.0	30.5	30.6	32.9	30.5	29.7	29.6	29.0
Aug 80	30.4	29.8	30.4	30.9	29.8	30.6	29.6	29.3	29.4
Sep 80	32.4	29.7	29.8	30.8	29.7	30.1	29.7	29.4	29.5
Oct 80	32.3	29.9	30.2	31.0	29.9	30.1	30.0	29.5	29.6
Nov 80	32.5	29.7	30.5	31.1	29.9	30.4	29.8	30.1	29.7
Dec 80	32.4	29.9	30.9	31.1	30.0	29.8	29.7	30.0	29.1
Jan 81	32.2	30.0	30.4	31.1	29.9	-	30.0	29.7	29.6
Feb 81	32.1	29.9	30.8	31.4	30.0	-	-	29.9	-
Mean	32.2	29.9	30.6	30.9	30.1	30.4	29.8	29.5	29.6
Std. Dev	1.00	0.17	0.40	0.21	0.84	0.29	0.16	0.45	0.27
High	33.0	30.2	31.1	31.4	32.9	30.7	30.0	30.1	29.6
Low	29.9	29.7	29.8	30.6	29.7	29.8	29.5	28.4	28.8

POUR POINT, °C (D 97)

Date	Virgin Stocks				Reference	Re-refined Stocks				
	A	B	C	D	I	E	F	G	H	J
Mar 80	-	-	-	-	-7	-	-	-	-	-
Mar 80	-7	-	-11	-15	-9	-12	-15	-11	-11	-9
Apr 80	-7	-7	-7	-14	-7	-9	-8	-9	-12	-9
May 80	-7	-8	-10	-17	-8	-9	-11	-9	-17	-9
Jun 80	-12	-9	-12	-18	-9	-9	-30	-9	-15	-12
July 80	-7	-8	-15	-13	-7	-9	-12	-9	-10	-12
Aug 80	-9	-6	-9	-15	-9	-9	-12	-12	-12	-12
Sep 80	-8	-5	-10	-15	-9	-10	-11	-11	-12	-9
Oct 80	-8	-6	-6	-18	-8	-10	-9	-8	-10	-12
Nov 80	-9	-9	-12	-18	-6	-9	-22	-9	-12	-10
Dec 80	-8	-7	-9	-16	-7	-14	-16	-11	-11	-15
Jan 81	-12	-10	-13	-19	-12	-	-10	-13	-14	-9
Feb 81	-8	-7	-9	-15	-7	-	-	-9	-11	-
Mean	-8.5	-7.4	-10.2	-16.1	-8.1	-10.0	-14.2	-10.0	-12.2	-10.7
Std. Dev	1.8	1.5	2.5	1.9	1.6	1.7	6.5	1.5	2.1	2.0
High	-7	-5	-6	-13	-6	-9	-8	-8	-10	-9
Low	-12	-10	-13	-19	-12	-14	-30	-13	-19	-15

CARBON RESIDUE, WT % (D 524)

Date	Virgin Stocks				Reference I	Re-refined Stocks				
	A	B	C	D		E	F	G	H	J
Mar 80	-	-	-	-	0.05	-	-	-	-	-
Mar 80	0.05	-	0.06	0.06	0.10	0.19	0.14	0.13	0.14	0.12
Apr 80	0.01	0.03	0.01	0.07	0.01	0.03	0.04	0.03	0.02	0.08
May 80	0.04	0.04	0.07	0.04	0.04	0.005	0.15	0.13	0.14	0.10
Jun 80	0.04	0.04	0.05	0.04	0.05	0.09	0.21	0.08	0.13	0.08
July 80	0.04	0.07	0.03	0.06	0.05	0.08	0.11	0.08	0.13	0.13
Aug 80	0.08	0.08	0.08	0.07	0.09	0.11	0.20	0.13	0.19	0.17
Sep 80	0.08	0.07	0.05	0.05	0.05	0.14	0.16	0.07	0.13	0.09
Oct 80	0.06	0.04	0.04	0.05	0.06	0.08	0.10	0.08	0.16	0.07
Nov 80	0.05	0.05	0.03	0.05	0.05	0.07	0.20	0.05	0.13	0.11
Dec 80	0.06	0.05	0.06	0.05	0.03	0.09	0.24	0.07	0.11	0.10
Jan 81	0.06	0.02	0.05	0.04	0.05	-	0.12	0.06	0.13	0.10
Feb 81	0.07	0.08	0.06	0.06	0.07	-	-	0.08	0.14	-
Mean	0.06	0.05	0.05	0.05	0.05	0.09	0.15	0.08	0.13	0.10
Std. Dev	0.02	0.02	0.02	0.01	0.02	0.05	0.06	0.03	0.04	0.03
High	0.08	0.08	0.08	0.07	0.10	0.19	0.24	0.13	0.19	0.17
Low	0.01	0.02	0.01	0.04	0.01	0.005	0.04	0.03	0.02	0.07

SULFATED ASH, WT% (D 874)

Date	Virgin Stocks				Reference I	Re-refined Stocks				
	A	B	C	D		E	F	G	H	J
Mar 80	-	-	-	-	0.00	-	-	-	-	-
Mar 80	0.00	-	0.00	0.00	0.00	0.02	0.00	0.00	0.00	0.00
Apr 80	0.00	0.00	0.00	0.00	0.00	0.01	0.02	0.07	0.00	0.00
May 80	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.03	0.00	0.00
Jun 80	0.00	0.04	0.00	0.00	0.00	0.02	0.13	0.01	0.00	0.00
July 80	0.01	0.00	0.0.00	0.00	0.00	0.01	0.01	0.00	0.00	0.00
Aug 80	0.00	0.00	0.00	0.00	0.00	0.00	0.03	0.00	0.00	0.00
Sep 80	0.00	0.00	0.00	0.00	0.00	0.00	0.03	0.01	0.00	0.00
Oct 80	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00
Nov 80	0.00	0.00	0.00	0.00	0.00	0.00	0.12	0.00	0.00	0.00
Dec 80	0.01	0.00	0.00	0.00	0.00	0.01	0.07	0.00	0.00	0.00
Jan 81	0.00	0.00	0.00	0.00	0.00	-	0.00	0.00	0.00	0.00
Feb 81	0.00	0.00	0.00	0.00	0.00	-	-	0.00	0.00	-
Mean	0.002	0.004	0.00	0.00	0.00	0.007	0.038	0.011	0.00	0.00
Std. Dev	0.004	0.012	0.00	0.00	0.00	0.008	0.048	0.021	0.00	0.00
H1gh	0.01	0.04	0.00	0.00	0.00	0.02	0.13	0.07	0.00	0.00
Low	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00

TOTAL ACID NUMBER (D 664)

Date	Virgin Stocks				Reference I	Re-refined Stocks				
	A	B	C	D		E	F	G	H	J
Mar 80	-	-	-	-	0.01	-	-	-	-	-
Mar 80	0.00	-	0.00	0.00	0.01	0.05	0.02	0.45	0.01	0.00
Apr 80	0.01	0.00	0.01	0.01	0.01	0.03	0.07	0.53	0.00	0.00
May 80	0.01	0.00	0.00	0.01	0.01	0.03	0.05	0.56	0.00	0.05
Jun 80	0.00	0.01	0.01	0.00	0.01	0.07	0.18	1.11	0.01	0.17
July 80	0.02	0.00	0.00	0.00	0.01	0.07	0.02	0.64	0.01	0.04
Aug 80	0.01	0.00	0.01	0.01	0.01	0.13	0.12	0.94	0.01	0.27
Sep 80	0.01	0.01	0.02	0.03	0.01	0.10	0.12	0.75	0.02	0.04
Oct 80	0.00	0.00	0.02	0.00	0.02	0.12	0.02	0.58	0.02	0.22
Nov 80	0.01	0.01	0.01	0.01	0.01	0.08	0.30	0.62	0.01	0.09
Dec 80	0.00	0.01	0.01	0.01	0.00	0.06	0.22	0.43	0.01	0.12
Jan 81	0.03	0.01	0.00	0.00	0.01	-	0.05	0.56	0.02	0.11
Feb 81	0.01	0.01	0.01	0.01	0.02	-	-	0.68	0.02	-
Mean	0.01	0.005	0.01	0.01	0.01	0.07	0.11	0.65	0.01	0.10
Std. Dev	0.01	0.005	0.01	0.08	0.005	0.03	0.09	0.20	0.01	0.09
High	0.03	0.01	0.02	0.03	0.02	0.13	0.30	1.11	0.02	0.27
Low	0.00	0.00	0.01	0.00	0.01	0.03	0.02	0.43	0.00	0.00

SAPONIFICATION NUMBER (D 94)

Date	Virgin Stocks				Reference I	Re-refined Stocks				
	A	B	C	D		E	F	G	H	J
Mar 80	-	-	-	-	0.16	-	-	-	-	-
Mar 80	0.05	-	0.05	0.02	0.17	0.60	0.15	1.46	0.09	0.17
Apr 80	0.06	0.23	0.03	0.04	0.14	0.52	0.22	2.10	0.14	0.40
May 80	0.06	0.24	0.02	0.06	0.17	0.46	0.16	1.91	0.15	0.15
Jun 80	0.08	0.18	0.11	0.02	0.14	0.59	0.55	2.37	0.27	0.23
July 80	0.06	0.21	0.40	0.03	0.09	1.03	0.12	1.12	0.09	0.15
Aug 80	0.16	0.20	0.06	0.04	0.23	0.74	0.28	*	0.39	0.61
Sep 80	0.16	0.24	0.05	0.02	0.13	1.15	0.27	2.35	0.18	0.16
Oct 80	0.16	0.27	0.04	0.04	0.15	0.82	0.06	2.39	0.35	0.45
Nov 80	0.07	0.27	0.06	0.00	0.15	0.80	0.77	*	0.29	0.30
Dec 80	0.10	0.28	0.05	0.03	0.14	0.70	0.49	1.64	0.20	0.31
Jan 81	0.10	0.25	0.04	0.02	0.12	-	0.14	1.82	0.12	0.36
Feb 81	0.22	0.31	0.09	0.06	0.20	-	-	2.26	0.18	-
Mean	0.11	0.24	0.08	0.03	0.15	0.74	0.29	1.94	0.20	0.30
Std. Dev	0.05	0.04	0.10	0.02	0.04	0.22	0.22	0.43	0.10	0.15
High	0.22	0.31	0.40	0.06	0.23	1.15	0.77	2.39*	0.39	0.61
Low	0.05	0.18	0.02	0.00	0.09	0.46	0.06	1.12	0.09	0.15

*Two Samples were too dark for accurate colorimetric determination

NITROGEN, ppm (Chemiluminescent)

Date	Virgin Stocks				Reference I	Re-refined Stocks			
	A	B	C	D		E	F	G	H
Mar 80	-	-	-	-	29	-	-	-	-
Mar 80	6	-	37	80	29	64	14	74	46
Apr 80	5	58	33	77	32	48	10	110	37
May 80	22	65	42	70	27	46	8	97	43
Jun 80	2	56	38	68	29	73	27	205	38
July 80	6	59	42	88	26	59	18	136	35
Aug 80	7	63	62	88	56	54	32	216	41
Sep 80	9	78	65	89	32	110	19	179	40
Oct 80	8	72	58	70	27	72	3	127	51
Nov 80	2	71	56	68	29	47	50	107	45
Dec 80	6	78	39	70	25	105	41	94	39
Jan 81	< 1	65	40	63	27	-	6	77	41
Feb 81	7	81	56	74	32	-	-	96	48
Mean	7	68	47	75	31	68	21	126	42
Std. Dev	6	9	11	9	8	23	15	48	5
High	22	81	65	89	56	110	50	216	51
Low	< 1	56	33	63	25	46	3	74	35

SULFUR, WT% (In-house X-Ray)

Date	Virgin Stocks				Reference I	Re-refined Stocks				
	A	B	C	D		E	F	G	H	J
Mar 80	-	-	-	-	0.66	-	-	-	-	-
Mar 80	0.04	-	0.32	0.06	0.66	0.24	0.14	0.28	0.23	0.23
Apr 80	0.04	0.36	0.36	0.07	0.64	0.22	0.14	0.30	0.21	0.25
May 80	0.04	0.39	0.36	0.06	0.65	0.23	0.14	0.29	0.22	0.23
Jun 80	0.04	0.38	0.39	0.06	0.64	0.24	0.20	0.33	0.20	0.23
July 80	0.04	0.40	0.49	0.06	0.65	0.25	0.15	0.25	0.24	0.20
Aug 80	0.06	0.40	0.49	0.06	0.64	0.24	0.17	0.29	0.22	0.26
Sep 80	0.02	0.41	0.51	0.06	0.66	0.28	0.16	0.30	0.24	0.19
Oct 80	0.07	0.42	0.51	0.05	0.67	0.26	0.12	0.31	0.24	0.25
Nov 80	0.06	0.39	0.47	0.04	0.67	0.25	0.19	0.23	0.20	0.24
Dec 80	0.07	0.41	0.37	0.05	0.66	0.29	0.17	0.26	0.22	0.25
Jan 81	0.07	0.41	0.47	0.04	0.67	-	0.13	0.27	0.23	0.22
Feb 81	0.07	0.46	0.43	0.04	0.67	-	-	0.26	0.20	-
Mean	0.05	0.40	0.43	0.05	0.66	0.25	0.16	0.28	0.22	0.24
Std. Dev	0.02	0.02	0.07	0.01	0.01	0.02	0.02	0.03	0.02	0.02
High	0.07	0.46	0.51	0.07	0.67	0.29	0.20	0.33	0.24	0.29
Low	0.02	0.36	0.32	0.04	0.17	0.22	0.12	0.23	0.20	0.19

ASTM COLOR (D 1500)

Date	Virgin Stocks				Reference I	Re-refined Stocks				
	A	B	C	D		E	F	G	H	J
Mar 80	-	-	-	-	2.5	-	-	-	-	-
Mar 80	1.5	-	1.0	0.5	2.5	3.0	4.5	5.0	6.0	4.5
Apr 80	1.5	1.5	0.5	0.5	2.5	2.5	4.0	5.0	5.0	4.0
May 80	1.5	1.5	0.5	2.0	2.5	2.0	3.5	6.5	6.0	3.0
Jun 80	1.5	1.5	0.5	0.5	2.5	3.0	4.5	7.0	6.0	3.5
July 80	2.0	1.5	0.5	0.5	2.5	2.5	3.5	7.5	6.0	2.0
Aug 80	1.5	1.5	0.5	1.0	2.5	2.0	4.0	8.0	8.0	4.5
Sep 80	1.0	1.0	1.0	1.0	3.0	3.0	3.5	6.0	6.0	3.5
Oct 80	2.0	2.0	1.5	1.0	2.5	3.5	4.0	6.5	7.5	5.0
Nov 80	2.0	2.0	1.0	0.5	2.5	2.5	4.5	5.5	6.0	4.5
Dec 80	2.0	1.5	0.5	1.0	2.5	3.0	4.5	5.5	6.5	5.0
Jan 81	2.0	1.5	0.5	0.5	2.5	-	3.5	4.5	6.0	4.5
Feb 81	2.0	2.0	0.5	0.5	2.5	-	-	5.5	6.0	-
Mean	1.7	1.6	0.7	0.8	2.5	2.7	4.0	6.0	6.2	4.0
Std. Dev	0.3	0.3	0.3	0.4	0.1	0.5	0.4	1.1	0.8	0.9
High	2.0	2.0	1.5	2.0	3.0	3.5	4.5	8.0	8.0	5.0
Low	1.0	1.0	0.5	0.5	2.5	2.0	3.5	4.5	5.0	2.0

AROMATICITY (UV), MONO-RING CARBON, WT% (In-house)

Date	Virgin Stocks				Reference I	Re-refined Stocks				
	A	B	C	D		E	F	G	H	J
Mar 80	-	-	-	-	4.0	-	-	-	-	-
Mar 80	3.2	-	4.3	2.7	4.1	3.3	4.2	3.9	3.6	3.6
Apr 80	3.0	3.1	4.4	2.6	3.9	3.2	4.2	3.5	3.9	3.9
May 80	3.0	3.2	4.5	2.7	4.0	3.1	4.1	3.5	3.8	3.8
Jun 80	2.9	3.2	4.2	2.2	3.9	3.1	3.4	3.4	3.6	3.6
July 80	3.0	3.3	4.7	2.8	3.9	3.3	4.0	3.5	3.8	3.8
Aug 80	3.9	3.5	5.1	2.6	4.1	3.4	4.2	3.6	4.0	4.0
Sep 80	3.8	4.0	5.0	2.8	4.0	3.3	4.6	3.6	3.5	3.5
Oct 80	3.7	3.3	4.9	2.5	4.0	3.0	3.9	3.6	3.9	3.9
Nov 80	3.5	3.5	4.8	2.4	3.9	3.5	3.8	3.6	4.0	4.0
Dec 80	3.9	3.4	4.9	2.6	3.9	3.3	4.0	3.6	3.7	3.7
Jan 81	3.9	3.4	4.9	2.4	4.0	3.2	4.1	3.6	3.7	3.7
Feb 81	3.8	3.6	4.5	2.6	4.1	-	4.1	3.6	-	-
Mean	3.5	3.4	4.7	2.6	4.0	3.9	3.2	4.0	3.6	3.8
Std. Dev	0.4	0.2	0.3	0.2	0.1	0.3	0.1	0.3	0.1	0.2
High	3.9	4.0	5.1	2.8	4.1	4.8	3.5	4.6	3.9	4.0
Low	2.9	3.1	4.2	2.2	3.9	3.6	3.0	3.4	3.4	3.5

AROMATICITY (UV), DI-RING CARBON, WT% (In-house)

Date	Virgin Stocks			Reference I	Re-refined Stocks					
	A	B	C	D	E	F	G	H	J	
Mar 80	-	-	-	-	1.6	-	-	-	-	-
Mar 80	1.2	-	1.1	0.7	1.7	1.7	2.3	1.7	2.1	2.1
Apr 80	1.1	1.0	1.2	0.7	1.6	1.4	2.1	1.6	2.0	2.0
May 80	1.2	1.1	1.2	0.7	1.6	1.4	2.2	1.6	1.6	1.6
Jun 80	1.1	1.1	1.2	0.6	1.6	2.0	2.6	1.4	1.8	1.8
July 80	1.2	1.1	1.6	0.7	1.5	1.5	2.1	1.5	2.0	2.0
Aug 80	1.5	1.3	1.7	0.7	1.7	1.6	2.3	1.6	1.9	1.9
Sep 80	1.5	1.4	1.8	0.7	1.6	1.6	2.3	1.5	1.8	1.8
Oct 80	1.5	1.2	1.7	0.6	1.6	1.4	2.1	1.6	1.8	1.8
Nov 80	1.4	1.2	1.6	0.6	1.6	1.4	1.9	1.6	2.1	2.1
Dec 80	1.7	1.3	1.6	0.7	1.6	1.7	2.0	1.6	1.7	1.7
Jan 81	1.6	1.2	1.6	0.3	1.6	-	2.2	1.6	1.7	1.7
Feb 81	1.6	1.3	1.3	0.6	1.7	-	1.9	1.7	-	-
Mean	1.4	1.2	1.5	0.6	1.6	1.6	2.2	1.6	1.9	1.9
Std. Dev	0.2	0.1	0.2	0.1	0.1	0.2	0.2	0.1	0.2	0.2
High	1.7	1.4	1.8	0.7	1.7	2.0	2.6	1.7	2.1	2.1
Low	1.1	1.0	1.1	0.3	1.5	1.4	1.9	1.4	1.6	1.6

AROMATICITY (IIV), TRI-RING CARBON, WT% (In-house)

Date	Virgin Stocks				Reference I	Re-refined Stocks				
	A	B	C	D		E	F	G	H	J
Mar 80	-	-	-	-	0.3	-	-	-	-	-
Mar 80	0.1	-	0.1	0	0.3	0.2	0.2	0.3	0.3	0.4
Apr 80	0.1	0.1	0.1	0.1	0.2	0.2	0.2	0.3	0.2	0.3
May 80	0.1	0.1	0.1	0.1	0.2	0.2	0.2	0.3	0.2	0.3
Jun 80	0.1	0.1	0.1	0.1	0.2	0.2	0.3	0.4	0.2	0.3
July 80	0.1	0.1	0.2	0.1	0.2	0.2	0.2	0.3	0.2	0.3
Aug 80	0.1	0.1	0.1	<0.1	0.3	0.2	0.2	0.4	0.2	0.3
Sep 80	0.1	0.1	0.1	<0.1	0.2	0.2	0.2	0.3	0.2	0.3
Oct 80	0.1	0.1	0.2	<0.1	0.2	0.2	0.2	0.3	0.2	0.3
Nov 80	0.1	0.1	0.1	<0.1	0.3	0.2	0.2	0.3	0.2	0.4
Dec 80	0.2	0.1	0.1	<0.1	0.2	0.2	0.2	0.3	0.2	0.3
Jan 81	0.2	0.1	0.1	<0.1	0.2	-	0.2	0.3	0.2	0.3
Feb 81	0.2	0.1	0.1	<0.1	0.3	-	-	0.3	0.2	-
Mean	0.1	0.1	0.1	<0.1	0.2	0.2	0.2	0.3	0.2	0.3
Std. Dev	0.04	0.00	0.04	*	0.05	0.00	0.03	0.04	0.03	0.04
High	0.2	0.1	0.2	0.1	0.3	0.2	0.3	0.4	0.3	0.4
Low	0.1	0.1	0.1	0.0	0.2	0.2	0.2	0.3	0.2	0.3

*Cannot be calculated because values reported as <0.1

8C BOILING POINT DISTRIBUTION (1% PT), °C (D 2887 Modified)

Date	Virgin Stocks				Reference I	Re-refined Stocks				
	A	B	C	D		E	F	G	H	J
Mar 80	-	-	-	-	355	-	-	-	-	-
Mar 80	337	-	305	372	376	299	305	274	328	316
Apr 80	342	363	296	367	359	307	296	275	287	305
May 80	339	391	301	362	365	301	299	279	322	302
Jun 80	339	386	316	362	373	314	292	275	335	308
July 80	342	387	302	364	360	298	290	285	323	327
Aug 80	342	381	309	362	361	306	311	286	332	334
Sep 80	343	354	297	358	358	297	301	284	325	337
Oct 80	350	389	302	372	364	319	314	290	334	333
Nov 80	340	308	291	366	356	306	289	280	319	337
Dec 80	341	387	299	353	360	304	297	280	317	348
Jan 81	348	376	311	334	362	-	294	276	326	336
Feb 81	347	396	322	362	362	-	-	298	320	-
Mean	342	374	304	361	362	305	299	282	322	326
Std. Dev	4	25	9	10	6	7	8	7	13	15
High	350	396	322	372	376	319	314	298	335	348
Low	337	308	291	334	355	297	289	274	287	302

GC BOILING POINT DISTRIBUTION (5% PT), °C (D 2887 Modified)

Date	Virgin Stocks				Reference I	Re-refined Stocks				
	A	B	C	D		E	F	G	H	J
Mar 80	-	-	-	-	388	-	-	-	-	-
Mar 80	357	-	355	397	398	343	369	322	367	355
Apr 80	390	410	349	396	390	347	359	319	361	358
May 80	358	416	353	393	397	343	364	324	361	355
Jun 80	359	417	371	392	406	359	356	317	370	359
July 80	360	419	349	393	390	345	360	337	364	369
Aug 80	361	420	354	393	393	347	369	334	371	369
Sep 80	356	410	348	393	393	345	368	338	364	371
Oct 80	361	425	352	403	397	360	374	343	372	366
Nov 80	356	376	343	396	390	348	362	343	360	373
Dec 80	354	421	354	397	391	349	366	346	360	378
Jan 81	360	417	364	396	392	-	366	326	367	369
Feb 81	360	418	353	396	392	-	-	350	362	-
Mean	361	414	354	395	393	349	365	335	365	366
Std. Dev	9	13	7	3	5	6	5	11	4	8
High	390	425	371	403	406	360	374	350	372	378
Low	354	376	343	392	387	343	356	317	360	355

GC BOILING POINT DISTRIBUTION (10% PT), °C (D 2887 Modified)

Date	Virgin Stocks			Reference I	Re-refined Stocks					
	A	B	C	D	E	F	G	H	J	
Mar 80	-	-	-	-	402	-	-	-	-	-
Mar 80	367	-	379	411	410	391	354	384	373	
Apr 80	404	426	377	410	404	383	347	380	380	
May 80	367	429	381	410	410	385	355	379	377	
Jun 80	368	431	397	406	419	383	344	385	380	
July 80	370	431	375	406	402	384	361	380	388	
Aug 80	369	435	379	407	408	390	362	388	385	
Sep 80	364	429	373	409	407	392	363	381	385	
Oct 80	369	440	378	417	410	395	368	389	381	
Nov 80	365	413	367	408	402	383	365	378	388	
Dec 80	362	435	378	412	404	387	368	377	392	
Jan 81	368	430	388	411	405	385	353	383	384	
Feb 81	367	430	375	409	405	-	367	377	-	
Mean	370	430	379	410	406	387	359	382	383	
Std. Dev	11	7	8	3	5	4	8	4	5	
High	404	440	397	412	419	395	368	389	392	
Low	362	413	367	406	399	383	347	377	373	

GC BOILING POINT DISTRIBUTION (50% PT), °C (D 2887 Modified)

Date	Virgin Stocks			Reference, I	Re-refined Stocks					
	A	B	C	D	E	F	G	H	J	
Mar 80	-	-	-	-	453	-	-	-	-	-
Mar 80	421	-	447	462	461	467	434	454	440	
Apr 80	457	477	451	461	457	459	427	450	452	
May 80	422	470	450	462	461	458	434	450	454	
Jun 80	423	478	461	460	467	462	431	453	448	
July 80	421	475	451	456	449	462	430	448	458	
Aug 80	423	482	451	462	458	463	438	462	446	
Sep 80	417	483	448	467	458	474	431	450	440	
Oct 80	424	492	455	471	462	473	444	461	440	
Nov 80	422	477	440	456	449	455	428	446	449	
Dec 80	417	483	444	461	453	459	431	445	450	
Jan 81	420	473	448	460	453	454	424	451	440	
Feb 81	414	476	432	454	450	-	424	437	-	
Mean	423	479	448	461	456	462	431	451	447	
Std. Dev	11	6	7	5	7	7	6	7	6	
High	457	492	461	471	467	474	444	462	458	
Low	414	470	432	454	443	454	424	437	440	

GC BOILING POINT DISTRIBUTION (90% PT), °C (D 2887 Modified)

Date	Virgin Stocks				Reference	Re-refined Stocks				
	A	B	C	D	I	E	F	G	H	J
Mar 80	-	-	-	-	502	-	-	-	-	-
Mar 80	480	-	493	513	511	474	572	500	542	504
Apr 80	511	532	501	512	511	476	561	492	539	522
May 80	482	505	493	509	509	472	555	503	546	538
Jun 80	482	522	500	510	513	489	560	504	551	518
July 80	481	512	498	501	494	485	569	502	548	539
Aug 80	482	524	494	513	504	477	573	513	565	510
Sep 80	478	531	493	519	504	497	574	497	544	502
Oct 80	487	548	503	532	513	504	>575	515	580	499
Nov 80	506	524	487	503	495	484	567	491	555	519
Dec 80	481	528	485	505	501	494	>575	490	546	520
Jan 81	483	512	488	505	503	-	573	491	551	510
Feb 81	477	526	475	503	498	-	-	492	528	-
Mean	486	524	492	510	504	485	567	500	550	516
Std. Dev	11	12	8	9	7	11	7	9	13	13
High	511	548	503	532	513	504	>575	515	580	539
Low	477	505	475	501	494	472	555	491	528	499

ASTM-NBS BASESTOCK CONSISTENCY SAMPLES

LUBTOT at 246°C

<u>Month</u>	<u>Oil I</u>	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>	<u>E</u>	<u>F</u>	<u>G</u>	<u>H</u>	<u>J</u>	<u>K</u>
Mar 80	29/36	32	X	36	20	14	50+	43	20	32	41
Apr	47	25	19	25	24	25	34	20	26	43	--
May	44	28	32	35	42	33	48	30	31	22	--
Jun	40	25	23	29	32	10	19	23	15	24	--
Jul	50	18	30	41	17	48	30	46	23	27	--
Aug	45	34	26	39	21	50+	28	36	20	29	--
Sep	34	31	18	28	24	35	14	46	25	32	--
Oct	39	32	24	38	28	33	40	15	24	48	--
Nov	42	42	28	40	27	19	16	50+	24	31	--
Dec	32	37	19	47	21	11	11	30	27	28	--
Jan 81	45	37	32	39	20	--	40	32	28	39	--
Feb	42	34	24	46	35	--	23	26	26	39	50+
Mar	30	39	14	38	25	--	--	--	24	41	--
Mean	40	32	24	37	26	28	29	33	24	34	--
Std.dev.	6.6	6.6	5.8	6.5	7.0	14.5	13.2	11.3	4.1	7.9	--
High	50	42	32	47	42	50+	50+	50+	31	48	--
Low	29	18	14	25	17	10	11	15	15	22	--

0 = clean.

-- = Not tested.

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